Designation: D 1274 – 95 (Reapproved 2000)

Standard Test Methods for Chemical Analysis of Pentachlorophenol[1]

This standard is issued under the fixed designation D 1274; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the chemical analysis of pentachlorophenol for use in the preservative treatment of wood.

1.1.1 Test Method D 38 covers the sampling of wood preservatives prior to testing.

1.2 The analytical procedures appear in the following order:

	Sections
Total Acidity	6 and 7
Alkali-Insoluble Material	8 and 9
Freezing Point	10

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 38 Test Methods for Sampling Wood Preservatives Prior to Testing[2]

D 1193 Specification for Reagent Water[3]

D 1272 Specification for Pentachlorophenol[2]

2.2 *Other Standard:*

AWPA A5 Standard Methods for Analysis of Oil-Borne Preservatives[4]

3. Summary of Test Methods

3.1 *Pentachlorophenol*—A measured sample is dissolved in methanol. The phenol is titrated with a standard solution of Sodium Hydroxide.

3.2 *Sodium Hydroxide Insoluble Matter*— A weighed sample is digested in 0.1 *N* NaOH and filtered. The residue is dried and weighed.

3.3 *Freezing Point*—A sample is heated in an enameled cup or nickel tube to 10°C above the expected melting point, then gradually cooled, and the melting point measured by a standardized thermometer.

4. Significance and Use

4.1 Pentachlorophenol for use in the preservative treatment of wood must conform with Specification D 1272.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

TOTAL ACIDITY

6. Reagents

6.1 *Sodium Hydroxide, Standard Solution* (0.1 *N*)—Dissolve 4.0 g of sodium hydroxide (NaOH) in 1000 mL of water.

6.1.1 *Standardization of Sodium Hydroxide Solution*—Transfer four 0.1 *N* potassium acid phthalate replicates (from 6.2), each of 25.00-mL volume, into 125-mL Erlenmeyer flasks. Add 3 to 5 drops of phenolphthalein indicator solution to each replicate. Titrate to a permanent faint pink end point with 0.1 *N* NaOH solution (from 6.1). Read the buret to the nearest estimated 0.01 mL. Record the volume of NaOH solution used for each replicate.

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[1] These test methods are under the jurisdiction of ASTM Committee D-7 on Wood and are the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

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These test methods are identical in substance with appropriate sections of the American Wood-Preservers' Association Standard Methods for Analysis of Oil-Borne Preservatives (A5-93). Acknowledgment is made to the American Wood-Preservers' Association for its development of the subject matter covered in this standard.

[2] *Annual Book of ASTM Standards*, Vol 04.10.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] Available from American Wood Preservers' Association, P.O. Box 286, Woodstock, MD 21163-0286.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6.2 *Potassium Acid Phthalate, Standard Solution* (0.1 *N*)—Weigh from 18 to 22 g of oven-dry primary standard grade potassium acid phthalate to the nearest 1 mg. Quantitatively transfer to a 1-L volumetric flask. Add water exactly to the mark. Mix well.

6.3 *Meta-Cresol Purple Indicator Solution*—Transfer 0.1 g *m*-cresol purple to a small mortar. Add 2.5 mL of 0.1 *N* NaOH solution and complete solution with the aid of a pestle. Add 100 mL of water.

6.4 *Phenolphthalein Indicator Solution* (5 g/L)—Dissolve 0.5 g of phenolphthalein in 50 mL of ethanol plus 50 mL of water.

6.5 *Methanol*.

7. Procedure

7.1 *Sample Preparation*—Weigh from 4 to 6 g of oven-dry sample to be analyzed to the nearest 1 mg. Quantitatively transfer to a 500-mL volumetric flask. Add methanol exactly to the mark. Mix well.

7.2 Transfer four replicates of the sample dissolved in methanol, each of 100.00-mL volume, into 250-mL flasks. Add 3 to 5 drops of *m*-cresol purple indicator solution. Titrate each replicate from a yellow color to a faint purple color with 0.1 *N* NaOH solution. Read the buret to the nearest estimated 0.01 mL. Record the millilitres of NaOH solution used for each replicate. Determine the reagent blank of four replicates of 100 mL of methanol plus 25 mL of water placed in 250-mL Erlenmeyer flasks.

7.3 *Calculations*:

7.3.1 Calculate the potassium acid phthalate normality as follows:

$$A = (0.004897) \times (B) \tag{1}$$

where:

A = normality of potassium acid phthalate solution (expressed to the nearest 0.0001 units), and

B = potassium acid phthalate from 6.2, g.

7.3.2 Calculate the sodium hydroxide normality as follows:

$$C = \frac{(A) \times (25.00)}{D} \tag{2}$$

where:

C = normality of NaOH solution (expressed to the nearest 0.0001 units),

A = normality of potassium acid phthalate from 7.3.1, and

D = NaOH solution used from 6.1.1, mL.

7.3.3 Calculate the percentage of pentachlorophenol:

$$E = \frac{(F - G) \times (C) \times (133.2)}{W} \tag{3}$$

where:

E = total acidity calculated as percent pentachlorophenol by weight (expressed to the nearest 0.1 units),

F = 0.1 N NaOH solution from 7.2, mL,

G = 0.1 N NaOH solution reagent blank from 7.2 (average of four determinations), mL,

C = normality of the NaOH solution from 7.3.2 (average of four determinations), and

W = sample from 7.1, g.

7.4 *Repeatability (95 % Confidence Limits)*—Individual values within a set of determinations should be no less than (0.9955) × (average for the set) and no greater than (1.0045) × (average for the set). The range of values (highest minus lowest) within a replicate set of four determinations should not exceed (0.0070) × (average for the set).

ALKALI-INSOLUBLE MATERIAL

8. Reagents

8.1 *Sodium Hydroxide Solution*—Dissolve 40 g of NaOH in water, and dilute to 1000 mL with water.

9. Procedure

9.1 Transfer approximately 1 g of the sample of pentachlorophenol, weighed to the nearest 1 mg, to a 250-mL beaker, and add 50 mL of NaOH solution and 50 mL of water. Warm to 60°C, and hold at 60 ± 5°C for 15 min.

9.2 Filter through a tared Gooch crucible having a medium-fiber asbestos mat, wash the residue with water, and dry at 100°C to constant weight.

9.3 *Calculation*—Calculate the percentage of alkaliinsoluble material as follows:

$$\text{Alkali} - \text{insoluble material, \%} = (A \times 100)/B \tag{4}$$

where:

A = residue remaining in the Gooch crucible, g, and

B = sample used, g.

FREEZING POINT

10. Procedure

10.1 Fill an enameled cup or a nickel tube of at least 1-in. (25-mm) diameter with sample. Melt in the direct flame of a Bunsen burner in a hood, stirring constantly with a standardized 160 to 195°C Thermometer.

10.2 When the molten material has a temperature about 10° above its expected melting point, allow it to cool while maintaining the constant stirring.

10.3 *Calculation*—As the freezing point is reached, heat of crystallization usually causes the temperature to increase slightly. Read the freezing point as the highest point at which the temperature remains constant for at least 15 s.

11. Precision and Bias

11.1 Data are not presently available to develop a precision and bias statement.

12. Keywords

12.1 chemical analysis; pentachlorophenol; preservative

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