



Standard Test Methods for Chemical Analysis of Fluor-Chrome-Arsenate-Phenol¹

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1. Scope

1.1 These test methods cover the determination of the chemical analysis of commercial fluor-chrome-arsenatephenol.

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
Fluorine (calculated as F)	6 to 8
Arsenic (calculated as As ₂ O ₅)	9 and 10
Chromium (calculated as CrO ₃)	11 and 12
Dinitrophenol or sodium pentachlorophenate	13 and 14

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 Testing²
- D 1033 Methods of Chemical Analysis of Chromated Zinc Chloride³
- D 1034 Specification for Fluor-Chrome-Arsenate-Phenol²
- D 1193 Specification for Reagent Water⁴

3. Summary of Test Methods

3.1 *Fluorine*—Fluorine is removed by steam distillation from an acidified sample. The distillate is neutralized and titrated with a standard solution of Thorium Nitrate. Fluorine is calculated as F.

3.2 *Arsenic*—A diluted sample is acidified with Hydrochloric and hypophosphorous acid to precipitate the arsenic. The arsenic is volatilized by heating in the presence of sulfuric acid,

which is then diluted with water and titrated with a standard solution of Potassium Bromate. Arsenic is calculated as As₂O₅.

3.3 *Chromium*—A diluted and chemically treated sample is reacted with an excess of ferrous ammonium sulfate. The unreacted ferrous ammonium sulfate is titrated with a standard solution of potassium dichromate to determine the consumed chromium. Hexavalent chromium is calculated as CrO₃.

3.4 *Phenol*—The sample is dissolved in water, acidified with sulfuric acid to free the phenolic compounds, and washed with ether to take up the phenols. The ether is evaporated and the residue measured as phenols.

4. Significance and Use

4.1 Fluor-Chrome-Arsenate-Phenol for use in the preservative treatment of wood must conform with Specification D 1034.

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.

FLUORINE

6. Apparatus

6.1 A suitable apparatus for the distillation of fluorine is shown in Fig. 1.

7. Reagents

7.1 *Potassium Permanganate Solution* (saturated).

7.2 *Sodium Alizarin Sulfonate Indicator*—Dissolve 0.05 g of sodium alizarin sulfonate in 100 mL of water.

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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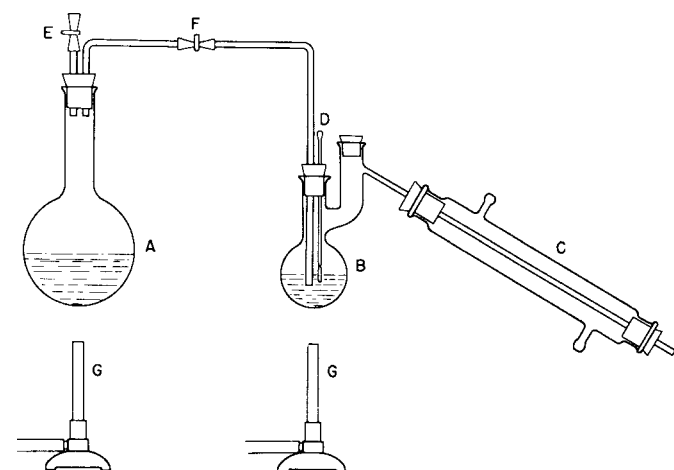
These methods are essentially the same as Sections 2, 5, 7, and 8 of the American Wood-Preservers' Association Standard Methods for Analysis of Water-Borne Preservatives and Fire-Retardant Formulations (A2-84). Acknowledgment is made to the American Wood Preservers' Association for its development of the subject matter covered in this standard.

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

³ Discontinued—See 1992 *Annual Book of ASTM Standards*, Vol 04.09.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

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.



A—Florence flask, 2000-mL (A number of stills can be operated from a single 2000-mL Florence flask)
B—Claissen flask, 250-mL

D—Thermometer
E and F—Pinchcocks
G—Bunsen burner

FIG. 1 Distillation Apparatus for Determination of Sodium Fluoride in Fluor-Chrome-Arsenate-Phenol

7.3 *Sodium Hydroxide Solution* (20 g/L)—Dissolve 2.0 g of NaOH in 100 mL of water.

7.4 *Hydrochloric Acid* (1 + 199).

7.5 *Buffer Solution*—Dissolve 9.448 g of monochloroacetic acid and 2.000 g of NaOH in 100 mL of water.

7.6 *Thorium Nitrate, Standard Solution* (0.1 N)—Dissolve 13.8 g of $\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}$ in water and dilute to 1 L. Standardize by titrating the fluorine obtained by distillation of 100 % natural cryolite ($3\text{NaF} \cdot \text{AlF}_3$) or NaF, using the procedure described in 8.

8. Procedure

8.1 Transfer a weighed portion of the sample, equivalent to about 0.025 g of F (Note 1), to a 250-mL Claissen flask, B, Fig. 1, containing eight to ten glass beads. Add 5 drops of KMnO_4 solution, adjust the total volume to 30 mL with water, and add 35 mL of H_2SO_4 .

NOTE 1—If this procedure is used for testing a fluor-chrome-arsenate-phenol treating solution, use an accurately measured volume of the filtered solution equivalent to the same amount of the element being determined.

NOTE 2—Hydrogen fluoride may react with glassware and lower the result. It is recommended that any available labware which would not be subject to fluoride attack, such as silica free glassware or plastic coated vessels, be used for the distillation and collection of the distillate.

8.2 Close the flask with a two-hole rubber stopper through which passes a thermometer and a 4-mm glass tube, both of which extend down into the solution. Use the 4-mm glass tube to connect the Claissen flask, B, Fig. 1, with a 2000-mL Florence flask, A, containing water, which is the source of steam for the ensuing distillation. Connect the Claissen flask, B, to the condenser, C, and with pinchcock F closed, distill until the boiling point reaches 140°C . Meanwhile, bring the water in flask A to a boil with pinchcock E open. When 140°C is reached in flask B, open pinchcock F to admit steam and close pinchcock E. Adjust the rate of distillation in flask B and

the flow of steam from flask A so as to maintain a constant volume of liquid in flask B and a boiling point of 140 to 145°C . Distill until 300 mL of distillate are collected.

8.3 To 100 mL of the distillate, add 8 drops of sodium alizarin sulfonate indicator solution. Adjust the acidity, using NaOH solution and HCl (1 + 199), until the solution is just acid and the pink color is discharged. Add 1 mL of the buffer solution and titrate with $\text{Th}(\text{NO}_3)_4$ solution to a persistent pink color.

8.4 *Calculation*—Calculate the percentage of fluoride as NaF, as follows:

$$F, \% = [(AB \times 0.018)/0.4C] \times 100 \quad (1)$$

where:

- A = $\text{Th}(\text{NO}_3)_4$ solution required for titration of the 100-mL aliquot of the sample, mL,
B = normality of the $\text{Th}(\text{NO}_3)_4$ solution, and
C = sample used, g.

ARSENIC

9. Reagents

9.1 *Hypophosphorous Acid* (50 %).

9.2 *Sulfuric Acid* (sp gr 1.84).

9.3 *Hydrochloric Acid* (sp gr 1.19).

9.4 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in water and dilute to 100 mL.

9.5 *Standard Potassium Bromate Solution* (0.1000 N)—Dissolve 2.784 g of KBrO_3 in water and dilute to 1 L in a volumetric flask.

10. Procedure

10.1 Transfer a weighed portion of the sample containing arsenic equivalent to about 0.15 g As_2O_5 (Note, 8.1) to a 250-mL wide-mouth Erlenmeyer flask and dilute with water to about 50 mL.

10.2 Add 50 mL of HCl and 20 mL of H_3PO_2 , mix thoroughly, and warm the solution on a steam bath until a precipitate forms. Boil gently for about 15 min. With the aid of suction, filter the hot solution through a 10-mL Gooch crucible containing a mat of medium-fiber, acid-washed asbestos, washing the flask and precipitate thoroughly with water.

10.3 Place the crucible containing the precipitate in the same flask in which the precipitation was carried out. Discard the filtrate. Pour 10 mL of H_2SO_4 into the flask and heat over an open flame in a hood, while agitating, until dense white fumes are evolved. Allow the flask and contents to cool and then add 100 mL of water very slowly and carefully, especially at first, as much heat is generated during this addition. Next add 5 mL of HCl and 2 drops of methyl orange indicator solution and titrate immediately with KBrO_3 solution. When the solution becomes colorless the end point has been reached.

10.4 *Calculation*—Calculate the percentage of arsenic in As_2O_5 , as follows:

$$\text{As}_2\text{O}_5, \% = A \times 0.5746/B \quad (2)$$

where:

- A = KBrO_3 solution required for titration of the sample, mL, and