



Standard Test Methods for Chemical Analysis of Chromated Copper Arsenate¹

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1. Scope

1.1 These test methods cover the chemical analysis of solid chromated copper arsenate and solutions of this material.

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

1.2 The analytical procedures occur in the following order:

	Sections
Pentavalent Arsenic (calculated as As ₂ O ₅)	7-9
Copper (calculated as CuO)	10-13
Hexavalent Chromium (calculated as CrO ₃)	14-16

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.* Specific precautionary statements are given in 8.2, 12.1.2, and in accordance with the safety precautions section of Test Method D 4278.

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 1035 Test Methods for Chemical Analysis of Fluor-Chrome-Arsenate-Phenol²
- D 1193 Specification for Reagent Water⁴
- D 1326 Methods for Chemical Analysis of Ammoniacal Copper Arsenate and Ammoniacal Copper Zinc Arsenate²
- D 1625 Specification for Chromated Copper Arsenate²
- D 1627 Methods for Chemical Analysis of Acid Copper Chromate²

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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The analytical methods and sampling procedures are substantially the same as those given in the American Wood-Preservers' Association Standard Methods for Analysis of Water-Borne Preservatives and Fire-Retardant Formulations (A2-82). Acknowledgment is made to the American Wood-Preservers' Association for its development of the subject matter covered in these test methods.

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.

D 4278 Test Method for Wet Ashing Procedure for Preparing Wood Samples for Inorganic Chemical Analysis²

3. Summary of Test Methods

3.1 Add 20 mL of tartaric acid solution to a 250-mL Erlenmeyer flask, then add 2 mL of the ACA concentrate. The resulting solution should become light blue-green. Twenty millilitres of sodium bicarbonate solution is then added and the solution will turn light blue. Two millilitres of the starch indicator is added. To this solution, one drop of iodine solution from a buret is added. If the solution turns a dark blue and remains, then the aeration is complete.

4. Significance and Use

4.1 These test methods test the completion of aeration which is used to convert trivalent arsenic to pentavalent arsenic.

5. Purity of 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.

6. Sampling

6.1 When the material to be sampled is a water solution, it shall be mixed to ensure uniformity and the sample shall be at least 0.45 L and preferably 0.9 L. The sample shall be representative and taken by a “thief” or other device. The sample shall be collected and stored in properly closed containers of glass or other suitable material.

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 *Anal. 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 When the material to be sampled consists of solids, a sample at least 2.3 kg in weight shall be taken from various points in the container or containers so that a representative sample is obtained. It shall be kept in an airtight container to prevent changes in composition by reason of moisture absorption or loss or chemical action of the air.

6.3 The analytical procedures given in these test methods specify samples containing between 0.1 and 1.0 g of the ingredient to be determined. If the sample is solid, unless it is dry and finely pulverized, it is preferable to weigh a larger sample than specified and dissolve this in a definite quantity of water from which aliquots containing the specified quantity may be taken for analysis. Prepared samples or solutions having a content of 10 to 20 g of solid preservative equivalent per litre are usually convenient. Samples of solution from working tanks or plant equipment shall be filtered at a working temperature immediately on obtaining and shall not be filtered at the time the analysis is performed. Should any precipitate or solid adhering to the container be present when the sample is analyzed, the solution and any such precipitate or solid shall be thoroughly intermixed before analysis in order to obtain a proper sample.

ARSENIC

NOTE 1—This procedure is essentially the same as the procedure for arsenic Method D 1326.

7. Reagents

7.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

7.2 *Hypophosphorous Acid* (50 %)—Concentrated hypophosphorous acid (H_3PO_2).

7.3 *Methyl Orange Indicator Solution* (0.1 g/L)—Dissolve 0.1 g of methyl orange in water and dilute to 1 L.

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

7.5 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8. Procedure

8.1 Transfer a measured portion of the sample containing arsenic equivalent to about 0.17 g of As_2O_5 to a 250-mL wide-mouth Erlenmeyer flask and dilute with water to about 50 mL. 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.

8.2 **Warning**—If the sample being analyzed is a wood sample digested with a perchloric acid mixture it now contains perchloric acid and a strong reducing agent, hypophosphorous acid. If it is evaporated too much, *it may explode with dangerous violence*. Do not boil longer than the specified time and cover the mouth of the Erlenmeyer flask with a small watchglass to minimize evaporation.

8.3 With the aid of suction, filter the hot solution, using a 10-mL Gooch crucible containing a mat of medium fiber, acid-washed asbestos, and washing the flask and precipitate thoroughly with water.

8.4 Place the crucible containing the precipitate in the flask in which the precipitation was carried out. Discard the filtrate. Pour 10 mL of H_2SO_4 into the flask and, while agitating, heat over an open flame in a hood until dense white fumes are evolved.

8.5 Allow the flask and contents to cool, and then add 100 mL of water very slowly and carefully, especially at first, since heat is generated during this addition. Next, add 5 mL of HCl and 2 drops of methyl orange indicator solution and titrate immediately with 0.1000 N KBrO_3 solution. When the solution becomes colorless, the end point has been reached.

9. Calculation

9.1 Calculate the percentage of pentavalent arsenic, As_2O_5 , as follows:

$$\text{As}_2\text{O}_5, \% = 0.5746 A/B \quad (1)$$

where:

A = 0.1000 N KBrO_3 solution required for titration of the sample, mL, and

B = sample used, g.

COPPER

NOTE 2—This procedure is essentially the same as the procedure for copper in Test Methods D 1326 and D 1627.

10. Reagents

10.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide ($\text{NH}_4\cdot\text{OH}$).

10.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

10.3 *Methanol*.

10.4 *Potassium Chlorate-Nitric Acid Mixture*—Dissolve 5 g of potassium chlorate (KClO_3) in 100 mL of concentrated nitric acid (HNO_3 , sp gr 1.42). Prepare this solution just before use and do not save any surplus solution for use later.

10.5 *Copper Foil or Shot*.

10.6 *Nitric Acid*, concentrated (sp gr 1.42).

10.7 *Urea Solution*, 5 %. Dissolve 5 g urea in 95 mL water.

10.8 *Acetic Acid*, glacial.

10.9 *Potassium Iodide Solution* (200 g/L)—Dissolve 200 g of potassium iodide (KI) in water and dilute to 1 L.

10.10 *Sodium Thiocyanate Solution* (200 g/L)—Dissolve 200 g of sodium thiocyanate (NaCNS) in water and dilute to 1 L.

10.11 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Dissolve 24.85 g of dry but not effloresced sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3\cdot 5\text{H}_2\text{O}$) and 1.0 g of sodium carbonate ($\text{Na}_2\cdot\text{CO}_3$) in water and dilute to 1 L.

10.11.1 Sodium thiosulfate solution prepared in accordance with 10.11 is usually close enough to 0.1 N and stable enough to give reasonable service. However, on standing, particularly at elevated laboratory temperatures, the titer of the solution may change. Therefore it is desirable to standardize the solution.

10.12 For standardization of the 0.1 N sodium thiosulfate solution, dissolve in a 250-mL Erlenmeyer flask an accurately weighed portion of pure copper foil or shot (about 0.25 g) in 10 mL of concentrated nitric acid. Evaporate the solution until