



# Standard Test Methods for Chemical Analysis of Ammoniacal Copper Quat, Type B (ACQ-B)<sup>1</sup>

This standard is issued under the fixed designation D 5584; 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 ( $\epsilon$ ) 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 commercial solutions of ammoniacal copper quat Type B (ACQ-B).

1.2 The analytical procedures appear in the following order:

- Ammonia
- Quat (Didecyltrimethylammonium chloride)
- Copper (calculated as CuO)

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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 1193 Specification for Reagent Water<sup>2</sup>
- D 1628 Test Methods for Chemical Analysis of Chromated Copper Arsenate<sup>3</sup>
- D 5654 Specification for Ammoniacal Copper Quat Type B (ACQ-B)

### 2.2 A WPA Standards:

- AWPA A2-92 Standard Methods for Analysis of Waterborne Preservatives and Fire Retardant Formulations<sup>4</sup>
- AWPA A9-90 Standard Method for Analysis of Treated Wood and Treating Solutions by X-ray Spectroscopy<sup>4</sup>
- AWPA A11-83 Standard Method for Analysis of Treated Wood and Treating Solutions by Atomic Absorption Spectroscopy<sup>4</sup>

## 3. Summary of Test Methods

3.1 *Ammonia in Solution*—Ammonia is freed from a caustic solution of the sample by distillation and absorbed in a boric

acid solution forming ammonium borate. This solution is titrated against 0.2 N sulfuric acid. The normality of any unreacted sulfuric acid solution is then determined by titration with standardized NaOH solution.

3.2 *Quat in Solution*—The concentration of quaternary ammonium compounds in ACQ-B concentrate and working solutions can be determined by titration using a number of procedures. Two of the possible methods are provided here. The first involves a two-phase (chloroform/water) titration. Sodium lauryl sulfate is used as the titrant and methylene blue as the color indicator. The end point of the titration is indicated by a color change in the organic layer from colorless to light blue. The second procedure involves a single-phase titration. After an initial neutralization step, ACQ-B solutions are titrated against sodium tetrphenylborate using 2, 7-dichlorofluorescein as the color indicator. The end point is indicated by a solution color change from purple to green.

3.3 *Quat in Wood*—Two alternate test methods are provided. The first procedure is a two-phase titrimetric method similar to that used for ACQ-B solutions. A high performance liquid chromatography (HPLC) method is also available. In the HPLC procedure a treated wood sample is ground to pass a 30-mesh screen and then extracted with acidified ethanol. An aliquot of this extract is filtered and then analyzed using a HPLC equipped with a Partisil SCX ion exchange column and a UV detector set at 262 nm. Benzyltrimethylammonium chloride is added to the HPLC mobile phase to allow indirect UV detection of DDAC type quats.

3.4 *Copper in Solution or Wood*—A variety of methods is available for determining the copper content in ACQ-B solution concentrates, work solutions, and wood. X-ray fluorescence is the most practical method for most wood treatment operations. The procedures involved in this technique are described in AWPA Standard A 9-90. An alternative procedure uses atomic absorption spectroscopy as outlined in AWPA Standard A-11-83. Copper in solution can also be determined titrimetrically using the procedure described in Test Methods D 1628.

## 4. Significance and Use

4.1 Ammoniacal copper quat Type B for use in the preservative treatment of wood must conform with Specification D 5654.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-7 on Wood and are the direct responsibility of Subcommittee D07.06 on Treatment for Wood Products.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standard*, Vol 04.10.

<sup>4</sup> Available from the American Wood Preservers' Association, P.O. Box 286, MD 21163.

## 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.<sup>5</sup> 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.

### DETERMINATION OF AMMONIA IN AMMONIACAL COPPER QUAT TYPE B (ACQ-B) FORMULATIONS

## 6. Scope

6.1 This test method is suitable for the detection of ammonia in solution provided that the sample analyzed contains ammonia or ammonium in amounts approximating but not exceeding 0.15 g NH<sub>3</sub> or NH<sub>4</sub>.

## 7. Apparatus

7.1 The apparatus consists of a 500-mL Kjeldahl flask to which is attached a spray trap by means of a rubber stopper. The spray trap can be found under “Kjeldahl distillation apparatus” in equipment catalogs where it is referred to as a “bulb.” The trap returns liquid to the distillation flask and permits vapor to pass to a water-jacket condenser that directs the condenser vapor downwards, then through a condenser adaptor, into a 100-mL Erlenmeyer flask.

## 8. Reagents

8.1 *Bromocresol Green Indicator*, 0.1 % solution—Dissolve 1.0 g bromocresol green in 1.5 mL 0.1 normal sodium hydroxide and dilute to 100 mL with distilled water.

8.2 *Magnesium Oxide Powder*.

8.3 *Potassium Acid Phthalate*, primary standard grade.

8.4 *Boric Acid Solution*, 4 %—Dissolve 40 g boric acid in 960 mL distilled water.

8.5 *Phenolphthalein Indicator*, 1.0 % solution—Dissolve 1.0 g phenolphthalein in 10 mL ethyl alcohol (such as J. T. Baker, No. 9400 alcohol, reagent).

8.6 *Sulfuric Acid Solution*, 0.2 *N*—Place about 10 mL distilled water in a 1000-mL volumetric flask, add 6.6 mL concentrated sulfuric acid and cool to 20°C. Dilute to 1 L with distilled water. (See standardization procedure below.)

8.7 *Sodium Hydroxide Solution*, 0.2 *N*—Dissolve 8.1 g sodium hydroxide in CO<sub>2</sub>-free distilled water, cool to room temperature and dilute to 1 L with CO<sub>2</sub>-free distilled water.

## 9. Procedure

9.1 Assemble the apparatus as described above but do not connect the 500-mL Kjeldahl flask. Place approximately 75 mL of the boric acid solution in the 500-mL Erlenmeyer flask, add four to five drops of bromocresol green indicator, and position the Erlenmeyer flask so that the tip of the condenser adaptor just dips into the boric acid solution.

9.2 Place the sample for analysis in the Kjeldahl flask. Dilute with distilled water to a volume of about 200 mL. Add a few glass beads to prevent bumping. Add 5.0 g of magnesium oxide and immediately attach the flask to the rest of the apparatus by means of the rubber stopper on the spray trap.

9.3 After making sure that all connections are tight, and the tip of the condenser adaptor is just below the surface of the boric acid solution, commence heating the contents of the Kjeldahl flask.

9.4 Distill off about 150 mL of liquid. Adjust the height of the Erlenmeyer flask throughout the distillation so that the tip of the condenser adaptor is always under, but near, the surface of the boric acid solution in the receiving vessel.

9.5 When the distillation is complete, lower the receiving vessel and remove the heat source. Wash down the condenser tube and adaptor into the receiving vessel, using distilled water.

9.6 Titrate the ammonium borate solution so formed with standard 0.2 *N* sulfuric acid.

9.7 For standardization of sodium hydroxide solution, weigh two portions of potassium acid phthalate  $1.6000 \pm 0.1000$  g, transferring each to 500 mL Erlenmeyer flasks. Dissolve in 100 mL freshly boiled and cooled water, adding two drops phenolphthalein. Titrate with the sodium hydroxide solution until a faint permanent pink color appears. Duplicate titrations should yield normalities within 0.0005 *N*.

9.8 For standardization of the sulfuric acid solution, pipet exactly 25 mL of the sulfuric acid solution into a 250-mL Erlenmeyer flask. Add two drops of phenolphthalein indicator and titrate with the standardized sodium hydroxide solution until a faint permanent pink color appears. Duplicate titrations should agree to within 0.10 mL. Record the average.

## 10. Calculation

10.1 *Normality of Sodium Hydroxide:*

$$\frac{\text{g Potassium acid phthalate}}{\text{mL NaOH} \times 0.2042}$$

10.2 *Normality of Sulfuric Acid:*

$$\frac{\text{normality of NaOH} \times \text{mL NaOH}}{25}$$

10.3 *Percent Active Ingredient:*

$$\frac{\text{mL H}_2(\text{SO}_4)(\text{normality H}_2\text{SO}_4)(\text{Factor})}{\text{g of sample}}$$

10.4 *Active Ingredient and Factor:*

Active Ingredient	Factor
NH <sub>3</sub>	1.703
NH <sub>4</sub>	1.804

<sup>5</sup> *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.