Designation: D 5584 – 94 (Reapproved 2000)

Standard Test Methods for Chemical Analysis of Ammoniacal Copper Quat, Type B (ACQ-B)[1]

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 (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 commercial solutions of ammoniacal copper quat Type B (ACQ-B).

1.2 The analytical procedures appear in the following order:

Ammonia
Quat (Didecyldimethylammonium 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[2]

D 1628 Test Methods for Chemical Analysis of Chromated Copper Arsenate[3]

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[4]

AWPA A9-90 Standard Method for Analysis of Treated Wood and Treating Solutions by X-ray Spectroscopy[4]

AWPA A11-83 Standard Method for Analysis of Treated Wood and Treating Solutions by Atomic Absorption Spectroscopy[4]

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 tetraphenylborate 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.

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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 Treatment for Wood Products.

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

[3] *Annual Book of ASTM Standard*, Vol 04.10.

[4] 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.[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.

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_3 or NH_4.

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_2-free distilled water, cool to room temperature and dilute to 1 L with CO_2-free distilled water.

[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.

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 ± 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*:

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

10.2 *Normality of Sulfuric Acid*:

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

10.3 *Percent Active Ingredient*:

$$\frac{mL\ H_2\ (SO_4)(\text{normality } H_2SO_4)(\text{Factor})}{\text{g of sample}}$$

10.4 *Active Ingredient and Factor*:

Active Ingredient	Factor
NH_3	1.703
NH_4	1.804

DETERMINATION OF QUATERNARY AMMONIUM COMPOUNDS IN ACQ-B SOLUTION BY TWO-PHASE TITRATION

11. Scope

11.1 This test method is applicable to the determination of quaternary ammonium compounds in ACQ-B working solutions by titration using sodium laurylsulfate as titrant and methylene blue as color indicator in a two-phase system. The end point of the titration is indicated by a color change in the organic layer from colorless to light blue.

12. Apparatus

12.1 *Microburet*, 10 mL capacity graduated in 0.02 mL increments or digital buret, 50 mL capacity.
12.2 *Glass Bottles*, 100 mL capacity with PTFE lined caps.
12.3 *Graduated Cylinder*, 25 mL.
12.4 *Analytical Balance*, 0.1 mg readability.
12.5 *Transfer Pipettes*.
12.6 *Volumetric Flasks*, 250 and 1000 mL.
12.7 *Erlenmeyer Flasks*, 125 mL.
12.8 *Beaker*, 250 mL.

13. Reagents

13.1 *Sodium Laurylsulfate*.
13.2 *Hyamine*, 1622.
13.3 *2′,7′-dichlorofluorescein*.
13.4 *Methylene Blue Indicator*.
13.5 *Sodium Sulfate*.
13.6 *Sulfuric Acid*, 0.43 *N*.
13.7 *Chloroform*.
13.8 *Isopropyl Alcohol*.

14. Solution Preparation

14.1 *Sodium Laurylsulfate* (0.005 *M*)—Dry several grams at 105°C to constant weight. Weigh out 1.44 g of dry sodium laurylsulfate (to the nearest mg) into a 250-mL beaker and dissolve in distilled water. Quantitatively transfer to a 1-L volumetric flask and dilute to volume with distilled water.

14.2 *Hyamine 1622* (0.005 *M*)—Dry to constant weight in an oven at 100°C. Weigh 0.580 to 0.585 g (to the nearest 0.1 mg) and dissolve in distilled water. Dilute to 250 mL in a volumetric flask.

NOTE 1—Hyamine 1622 is diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride.

14.3 *Methylene Blue Solution*—Weigh out 0.03 g of methylene blue and transfer to a 1-L volumetric flask. Weigh out 50 g of sodium sulfate and transfer to the 1-L volumetric flask. Dilute to volume with 0.43 *N* sulfuric acid.

14.4 *2′,7′-Dichlorofluorescein Color Indicator*—0.1 % in isopropyl alcohol.

15. Procedure

15.1 *Standardization of Sodium Laurylsulfate Solution*:
15.1.1 Weigh out 5 g (to the nearest 0.1 mg) Hyamine 1622 solution into a 125-mL Erlenmeyer flask.
15.1.2 Add 25 mL of distilled water and seven drops of 2’,7’-dichlorofluorescein indicator solution.
15.1.3 Titrate with sodium laurylsulfate solution to the color change from pink to yellow-green marked by the formation of a white precipitate.

15.2 Add 20 mL water, 20 mL chloroform and 2 mL methylene blue solution by graduated cylinder to the glass jar. Cap the jar and shake the mixture well. Weigh 3 g of sample solution (to the nearest 1 mg) into the tared jar and record the mass. Cap the jar and shake the mixture well. Titrate with the standardized (0.005 *M*) sodium laurylsulfate solution. The end point is where the chloroform layer (bottom) just begins to turn blue. If the aqueous layer turns white and the chloroform layer is blue then the end point has been exceeded. The detection limit under these conditions is a solution concentration of 0.001 % (10 ppm mass/mass) alkyl ammonium compound.

16. Calculation

16.1 *M1*:

$$hyamine\ solution = \frac{\text{hyamine mass (g)}}{(448.1)(0.25)}$$

16.2 *M2*:

$$sodium\ laurylsulfate = \frac{(\text{M1})(\text{g of } Hyamine)}{mL\ of\ sodium\ laurylsulfate}$$

$$\%alkyl\ ammonium\ compound = \frac{(\text{M2})(mL\ titrant)(\text{MW of AAC})(100)}{(1000)(\text{g } of\ sample)}$$

DETERMINATION OF QUATERNARY AMMONIUM COMPOUNDS IN ACQ-B SOLUTIONS BY SINGLE-PHASE TITRATION

17. Scope

17.1 This test method is applicable to the determination of didecyldimethyl ammonium chloride (DDAC) in ACQ-B working solutions by titration using sodium tetraphenylboron as titrant and 2′,7′-dichlorofluorescein as a color indicator. For ACQ concentrates a suitable dilution before the analysis is required. In the case of alkaline ACQ solutions the solution should be first neutralized. The end point of the titration will be indicated by a solution color change from purple-pink to lemon-green.

18. Apparatus

18.1 *Class A Buret*, 50 mL.
18.2 *Erlenmeyer Flask*, 125 mL.
18.3 *Analytical Balance*, 0.1 mg readability.
18.4 *Volumetric Pipette*, 5 mL.
18.5 *Volumetric Flask*, 250 mL.
18.6 *Graduated Cylinder*, 25 mL.

19. Reagents

19.1 *Sodium Tetraphenylboron*—Dissolve 0.865 g sodium (0.0025 *M*) tetraphenylboron in distilled water and dilute to 1 L in a volumetric flask.

19.2 *2′,7′-Dichlorofluorescein*, 0.1 % in isopropyl alcohol.

19.3 *Hyamine 1622*, 0.01 *M*—Dry Hyamine 1622 to constant weight in an oven at 100°C. Weigh 1.16 to 1.17 g (to nearest 0.1 mg) and dissolve in distilled water. Dilute to 250 mL in a volumetric flask.

20. Standardization of Sodium Tetraphenylboron Solution

20.1 Pipet 5 mL of Hyamine 1622 solution into a 125-mL Erlenmeyer flask.

20.2 Add 25 mL distilled water and seven drops of 2′,7′-dichlorofluorescein indicator solution.

20.3 Slowly titrate with sodium tetraphenylboron solution.

20.4 End point is a lemon-green color solution and it is common for a precipitate to form just prior to the end point.

21. Procedure

21.1 Weigh 2 g (to nearest 0.1 mg) of treatment solution into a 125-mL Erlenmeyer flask.

21.2 Add 25 mL of distilled water into the solution.

21.3 Neutralize the solution by drop-wise addition of concentrated phosphoric acid with mixing to approximately pH 7 as indicated by the disappearance of the deep blue color and formation of a very light blue precipitate.

21.4 Add 14 drops of 2′,7′-dichlorofluorescein color indicator to the solution. The solution will then have a light pink color.

21.5 Titrate with sodium tetraphenylboron (STPB) to the lemon-green color end point and record the volume (mL) of sodium tetraphenylboron used.

22. Calculation

22.1 *Hyamine Concentration*:

$$M\ Hyamine = \frac{Hyamine\ mass\ (g)}{448.1 \times 0.25\ L}$$

22.2 *Sodium Tetraphenylboron Concentration*:

$$M(STPB) = \frac{(M\ Hyamine) \times (mL\ Hyamine)}{mL\ STPB\ added}$$

22.3 *% DDAC in Solution*:

$$\%\ DDAC = \frac{100 \times (mL\ STPB\ ) \times (M\ STPB) \times 362.1}{1000 \times (sample\ mass,\ g)}$$

DETERMINATION OF QUATERNARY AMMONIUM COMPOUNDS IN WOOD BY TWO-PHASE TITRATION

23. Scope

23.1 This test method is used to determine the concentration of didecyldimethylammonium chloride in treated wood. This test method is not compound specific, the total equivalence of cationic surfactants is determined. This procedure is intended for routine quality control in wood treatment and is not suitable for determination of trace levels of quaternary ammonium compounds in wood.

24. Summary of Test Method

24.1 An anionic surfactant (sodium lauryl sulfate) is titrated with a standard cationic surfactant (Hyamine 1622) in a chloroform/water two-phase system. A cationic dye (dimidium bromide) and an anionic dye (erioglaucine) are used in the system to visually determine the end point. When the anionic surfactant is in excess, a pink chloroform soluble complex is formed with the cationic dye. In the titration, Hyamine 1622 forms a more stable complex with the anionic surfactant and displaces the cationic dye from the anionic surfactant/dye complex and from the chloroform phase. The first excess of Hyamine 1622 reacts with the anionic dye (erioglaucine) to form a blue colored chloroform soluble complex.

24.2 Quaternary ammonium compounds are cationic surfactants. The determination is based on a back titration. A sample aliquot is added to the sodium lauryl sulfate solution. The excess sodium lauryl sulfate is determined by titration with Hyamine 1622.

25. Equipment

25.1 *Microburet*, 10 mL graduated in 0.02 mL increments.

25.2 *Separatory Funnel*, 125 mL glass stopper with PTFE stopcock.

25.3 *Ultrasonic Bath*.

25.4 *Screw Cap Vials*, 20 mL with PTFE-lined caps.

26. Reagents

26.1 *Sodium Lauryl Sulfate*, 0.004 *M*—Weigh between 1.14 to 1.16 g of sodium lauryl sulfate into a 250-mL beaker and dissolve in distilled water (100 mL). Add a drop of triethanolamine and quantitatively transfer to a 1-L volumetric flask and dilute to volume with distilled water.

26.2 *Hyamine 1622 (p-tertoctylphenoxyethoxyethyldimethylbenzyl ammonium chloride)* 0.004 *M*—Dry the Hyamine 1622 to a constant weight in an oven at 105°C. Weigh between 1.75 and 1.85 g (to the nearest 0.1 mg) into a beaker and dissolve in distilled water. Transfer quantitatively to a 1-L volumetric flask. Dilute to volume with distilled water.

26.3 *Erioglaucine*.

26.4 *Dimidium Bromide* (3,8-Diamino-5-methyl-6-phenylphenanthridium bromide).

26.5 *95 % Denatured Ethanol*, reagent grade.

26.6 *Mixed Indicator Stock Solution*— Add 0.50 g of dimidium bromide and 0.25 g of erioglaucine to a 100-mL beaker. Dissolve in 50 mL 50:50 water denatured 95 % ethanol solution and transfer quantitatively to a 250-mL volumetric flask with ethanol rinsing. Dilute to volume with denatured ethanol.

26.7 *Acid Indicator Solution* % Add 200 mL distilled water and 80 mL of mixed indicator solution into a 2-L volumetric flask. Add 80 mL of 2.5 *M* H_2SO_4 and dilute to volume with distilled water. Store in an amber bottle out of direct sunlight.

26.8 *Chloroform*, reagent grade.

26.9 *Hydrochloric Acid* 36 %, reagent grade.

26.10 *Extraction Solution*, 0.1 *N* HCl— Add 8.33 g HCl to a 1-L volumetric flask and dilute to volume with denatured ethanol.

27. Sample Extraction

27.1 Grind the air-dried wood samples in to pass a 30-mesh screen in a Wiley mill. Transfer a 1.5-g, to nearest 0.001 g, sample of oven-dried wood meal to a 20-mL screw cap vial. Add 15 mL of the 0.1- *N* HCl extraction solution by volumetric pipet and seal tightly with the PTFE lined caps. Place in an

ultrasonic bath and agitate for 3 h. Allow the mixture to cool and the wood meal to settle (centrifuge if necessary) before analysis.

28. Standardization of Sodium Lauryl Sulfate

28.1 Add 5 mL of the 0.004 *M* sodium lauryl sulfate solution by volumetric pipet to a 125-mL separatory funnel.

28.2 Add 20 mL distilled water, 15 mL of chloroform and 10 mL of the acid indicator solution by graduated cylinder.

28.3 Titrate with the 0.004 *M* Hyamine 1622 solution. Stopper the separatory funnel and shake after each addition of titrant. Rinse the stopper with distilled water to avoid sample loss as it is removed.

28.4 The chloroform layer will be colored pink before the end point. As the end point is approached the chloroform/water emulsion will break more readily and the aqueous phase color changes from gray to green. Continue titration with smaller additions until pink color begins to change.

28.5 The end point is taken at the point where the pink color is completely discharged and the chloroform layer is a faint grey-blue color. With excess Hyamine the chloroform layer becomes blue. Record this volume as V_o.

29. Sample Analysis

29.1 Titrate a 3-mL aliquot of the extraction solution with 0.05 N NaOH to the phenolphthalein end point. Use this predetermined amount to neutralize the wood extracts.

29.2 Transfer a 3-mL aliquot of the wood extract from 5.1 by volumetric pipet to a 125-mL separatory funnel containing 20 mL distilled water. Add the predetermined amount of 0.05 *N* NaOH by graduated pipet. Add 15 mL chloroform and 10 mL of acid indicator solution by graduated cylinder. Add 5 mL of the 0.004 *M* sodium lauryl sulfate solution by volumetric pipet.

29.3 Shake the separatory funnel. At this point the chloroform layer should be pink. If the chloroform layer is blue, insufficient sodium lauryl sulfate is present. Another 5 mL of sodium lauryl sulfate can be added, but this will need to be taken into account in the calculations.

29.4 The excess sodium lauryl sulfate is titrated with 0.004 *M* Hyamine 1622 to the gray-blue end point as in 6.3 through 6.5. Record the volume as *V*.

30. Calculation

30.1 *Molarity Hyamine 1622:*

$$\frac{\textit{mass hyamine } 1622\ (g)}{448.1}$$

$$\%\ wt\ quaternary\ ammonium\ compound = \frac{(V_o - V)\ (\textit{Molarity Hyamine } 1622)(\textit{MW } \text{quat})(0.5)}{\textit{sample mass } (\text{g})}$$

where:

V_o = volume (mL) of Hyamine 1622 required in blank titration,

V = volume (mL) of Hyamine 1622 required in sample titration,

Molecular weight quat = molecular weight of quaternary ammonium compound, and

Molecular weight of didecyldimethyl ammonium chloride = 362.08.

HPLC METHOD FOR DIDECYLDIMETHYLAMMONIUM CHLORIDE DETERMINATION IN TREATED WOOD

31. Scope

31.1 This test method is applicable to the determination of didecydimethyl ammonium chloride (DDAC) in wood using high performance liquid chromatography (HPLC) with indirect ultraviolet (UV) detection following extraction. The chromatographic peaks appear as troughs or negative peaks, Monovalent cations produce an interference in the chromatogram.

32. Apparatus

32.1 *HPLC System*—Perkin-Elmer Model 410 pump with 10 µL loop injector, LC95 UV/Vis detector set at 262 nm and LCI-100 integrator, or equivalent.

32.2 *Whatman SCX Cation Exchange Column*, with particle size of 5 µm, column length of 100 mm and inside diameter of 4.6 mm or equivalent.

32.3 *pH Meter*.

32.4 *Screw Cap Vials*, 25 mL, with PTFE lined caps.

32.5 *HPLC Syringe Filters*, 045 µm PTFE.

32.6 *Ultrasonic Bath*.

32.7 *Analytical Balance*, 0.1 mg readability.

32.8 *Class A Volumetric Pipet*, 20 mL.

33. Reagents

33.1 *Methanol*, HPLC grade.

33.2 *Water*, HPLC grade.

33.3 *Denatured Ethanol*, ACS reagent grade.

33.4 *Formic Acid*, ACS reagent grade.

33.5 *Benzyltrimethylammonium Chloride*, ACS reagent grade.

33.6 *Didecyldimethylammonium Chloride*, analytical standard.

33.7 *Acetic Acid*, ACS reagent grade.

33.8 *Extraction Solution:* Adjust the pH of the denatured ethanol to 5.0 ± 0.1 with formic acid.

33.9 *HPLC Mobile Phase*:

33.9.1 Mix HPLC grade water and HPLC grade methanol in a 1:5 ratio (v/v).

33.9.2 Add 0.75 g of benzyltrimethyl-ammonium chloride and 10.0 mL of acetic acid into a 1-L flask then add water/methanol solution to 1-L volume. Stir to fully dissolve. Filter through a 0.45-µm PTFE membrane filter.

34. Calibration

34.1 Perform a calibration with each analysis batch.

34.1.1 Equilibrate the HPLC system at a mobile phase flow rate of 3.0 mL/min before calibration and analysis.

34.1.2 Prepare didecyldimethyl ammonium chloride (DDAC) standards of 50, 100, 500, 1000 ppm in pH = 5 denatured ethanol for calibration.

34.1.3 Measure the chromatographic peak (retention time: 3.0 min) height of the standards from the base line. Alternatively use the peak heights determined by an integrator.

34.1.4 Plot the peak height or area versus concentrationand calculate the regression equation for calibration.

35. Procedure

35.1 Weigh 500 mg (to nearest 0.1 mg) of wood meal (outside diameter basis) sample (30 mesh) into a screw cap PTFE lined test vial.

35.2 Add 20.0 mL of extraction solution by volumetric pipet and screw the cap on tightly to prevent evaporation.

35.3 Immerse the vial half way in an ultrasonic bath solution and sonicate for 3 h. After completing the extraction, remove the vial from ultrasonic bath and allow to cool and settle before analysis.

36. Sample Analysis

36.1 Filter an aliquot of the sample extracts for injection into the HPLC system through a syringe filter.

36.2 Inject or use an autosampler to run the samples on the HPLC and measure the peak height of the sample peaks by direct measurement or determine using an integrator.

36.3 Calculate the DDAC concentration (ppm) in the extract from the peak height using the calibration regression equation.

36.4 Calculate the DDAC concentration (%) in wood using:

$$\%DDAC = \frac{(ppm\ DDAC\ in\ extract) \times 0.02\ L}{(sample\ mass\ g) \times 10}$$

37. Keywords

37.1 ammonia; ammoniacal copper quat Type B (ACQ-B); wood