

Designation: D 1326 – 94 (Reapproved 2000)

# Standard Test Methods for Chemical Analysis of Ammoniacal Copper Arsenate and Ammoniacal Copper Zinc Arsenate<sup>1, 2</sup>

This standard is issued under the fixed designation D 1326; 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 arsenate.
- 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
Ammonium bicarbonate (calculated as carbonate)	20 to 22
Ammonia (calculated as anhydrous ammonia)	6 to 9
Arsenic (calculated as arsenic pentoxide)	10 to 11
Copper (calculated as copper oxide)	13 to 16
Zinc (calculated as zinc oxide)	17 to 19

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<sup>3</sup>
- D 1193 Specification for Reagent Water<sup>4</sup>
- D 1325 Specification for Ammoniacal Copper Zinc Arsenate<sup>3</sup>
- D 3873 Test Method for Valency State of the Arsenic Component of Ammoniacal Copper Arsenate Solutions<sup>3</sup>
- 2.2 AWPA Standard:

AWPA A2 Standard Methods for Analysis of Waterborne Preservatives and Fire-Retardant Formulations<sup>5</sup>

#### 3. Summary of Test Methods

- 3.1 Ammonia—Ammonia is freed from a caustic solution of the sample by distillation and absorbed in a measured excess of sulfuric acid solution. The unreacted sulfuric acid solution is titrated with standardized NaOH solution to determine the ammonia reacted with sulfuric acid.
- 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 As<sub>2</sub> O<sub>5</sub>.
- 3.3 *Copper*—A measured sample is dissolved in hydrochloric acid and reacted with potassium iodide to complex the copper ion. The solution is then titrated with a standard solution of sodium thiosulfate. The copper is calculated as CuO.
- 3.4 Zinc—A diluted and chemically treated sample is treated with a standard solution of potassium ferrocyanide in the presence of an oxidation-reduction indicator (diphenylamine). The zinc is calculated as ZnO.

# 4. Significance and Use

4.1 Ammoniacal copper arsenate and ammoniacal copper zinc arsenate for use in the preservative treatment of wood must conform with Specification D 1325.

# 5. Purity of Reagents

5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such

<sup>&</sup>lt;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 Treatments for Wood Products.

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<sup>&</sup>lt;sup>2</sup> The analytical methods and sampling procedures are essentially the same as those given in the American Wood-Preservers' Assn. Standard Methods for Analysis of Water-Borne Preservatives and Fire-Retardant Formulations (A2-91). Acknowledgment is made to the American Wood-Preservers' Association for its development of the subject matter covered in this test.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 04.10.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>5</sup> Available from American Wood-Preservers' Association, P.O. Box 286, Wood-stock, MD 21163-0286.



specifications are available.<sup>6</sup> Other grades shall only be used when 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

#### **AMMONIA**

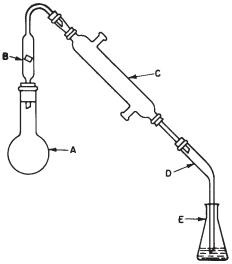
# 6. Apparatus

6.1 A typical apparatus for the distillation of ammonia is shown in Fig. 1.

# 7. Reagents

- 7.1 Methyl Purple or Methyl Red Indicator Solution—Suitable prepared solutions of methyl purple can be purchased. To prepare the methyl red indicator solution, dissolve 0.1 g of methyl red in 50 mL of alcohol (90 %), dilute to 100 mL with water, and filter if necessary.
- 7.2 Phenolphthalein Indicator Solution— Dissolve 1 g of phenolphthalein in 100 mL of alcohol.
- 7.3 Sodium Hydroxide Standard Solution (0.1 N)—Dissolve 4 g of NaOH in  $CO_2$ -free water and dilute to 1 L with  $CO_2$ -free water. Standardize as follows: Place 2 0.8000-g portions of potassium acid phthalate (National Bureau of Standards standard sample No. 84) in volumetric flasks, and add 100 mL of

<sup>&</sup>lt;sup>6</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.



A-Round-bottom flask, 500-mL

B—Distillation trap.

C-Condenser.

D-Delivery tube.

E-Erlenmeyer flask, 500-mL.

FIG. 1 Distillation Apparatus for Determination of Ammonia in Ammoniacal Copper Arsenate and Ammoniacal Copper Zinc Arsenate

freshly boiled water and 2 drops of phenolphthalein indicator solution to each flask. Titrate with the NaOH solution to the appearance of a faint, persistent pink color. The titrations should agree within 0.1 mL; record the average. Calculate the normality of the NaOH solution as follows:

Normality of NaOH solution = 
$$3.920/V$$
 (1)

where V = mL of NaOH solution required.

7.4 Sulfuric Acid, Standard (0.1 N)—To 100 mL of water in a 1000-mL volumetric flask, add 3.3 mL of  $\rm H_2~SO_4$  (sp gr 1.84); mix and cool to 20°C. Dilute to the mark with water. Standardize as follows: Pipet 25 mL of the  $\rm H_2~SO_4$  solution into a 500-mL Erlenmeyer flask, and add 100 mL of water and 2 drops of phenolphthalein indicator solution. Titrate with the 0.1 N NaOH solution to the appearance of a faint, persistent pink color. Calculate the normality of the  $\rm H_2~SO_4$  as follows:

Normality of 
$$H_2 SO_4 = VN/25$$
 (2)

where:

V = NaOH solution required, mL, and

N = normality of the NaOH solution.

7.5 Sodium Hydroxide Solution (100 g/L)—Dissolve 100 g of NaOH in water and dilute to 1 L.

# 8. Procedure

8.1 Transfer a portion of the sample containing not more than 0.15 g of NH $_3$  (Note 1) to the 500-mL distillation flask. Dilute with water to a volume of about 200 mL. Add a few glass beads to prevent bumping. Add 20 mL of NaOH solution (100 g NaOH/L) and immediately close the flask with a rubber stopper through which a spray trap has been inserted. The spray trap shall previously have been connected to a vertical condenser. Place 100 mL of 0.1 N H $_2$  SO  $_4$  in a 400-mL beaker. Adjust the tip of the condenser so that it just dips into the H $_2$  SO $_4$ . Add 4 to 5 drops of methyl purple or methyl red indicator solution to the H $_2$  SO $_4$ .

Note 1—If this procedure is used for testing a solution of treating strength, use an accurately measured volume of the filtered solution equivalent to the appropriate amount of the element to be determined.

- 8.2 After making sure all the connections are airtight, commence heating the mixture of sample and NaOH solution. Distill slowly at first, and finally boil rather vigorously. Distill off about 150 mL of solution. Adjust the height of the beaker throughout the distillation so that the tip of the condenser is always near the surface of the liquid in the receiving vessel. If the indicator starts to turn color, add a measured amount of additional 0.1 N H<sub>2</sub> SO<sub>4</sub>. When distillation is complete, turn out the flame and lower the receiving vessel. Wash down the condenser tube and its tip into the receiver.
  - 8.3 Titrate the excess acid with 0.1 N NaOH solution.

#### 9. Calculation

9.1 Calculate the percentage of ammonia as NH  $_3$ , as follows:

$$NH_3, \% = [(AB - CD) \times 1.703]/E$$
 (3)



where:

 $A = H_2 SO_4$  added to the receiving vessel, mL,

 $B = \text{normality of the H}_2 \text{ SO}_4$ ,

 $C = \text{NaOH solution used for titration of the excess H}_2 \text{SO}_4$ 

mL,

D = normality of the NaOH solution, and

E = sample used, g.

# **ARSENIC**

# 10. Reagents

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

10.2 Hypophosphorous Acid (50 %).

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

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

10.5 Sulfuric Acid (sp gr 1.84)—Concentrated sulfuric acid (H<sub>2</sub> SO<sub>4</sub>).

#### 11. Procedure

11.1 Transfer a portion of the sample containing arsenic equivalent to about 0.17 g As $_2$  O $_5$  (see Note 1) 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 $_3$  PO $_2$ , 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.

11.2 Place the crucible containing the precipitate in the flask in which the precipitation was carried out. Discard the filtrate. Pour 10 mL of  $\rm H_2~SO_4$  into the flask and while agitating, heat over an open flame in a hood 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, since heat is generated during this addition. Next, add 5 mL of HCl and 2 drops of methyl orange indicator and titrate immediately with KBrO<sub>3</sub> solution. When the solution becomes colorless, the end point has been reached.

# 12. Calculation

12.1 Calculate the percentage of arsenic as As  $_2$  O $_5$ , as follows:

$$As_2 O_5, \% = 0.5746 A/B \tag{4}$$

where:

A = KBrO<sub>3</sub> solution required for titration of the sample, mL, and

B = sample used, g.

# **COPPER**

# 13. Reagents

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

13.2 Potassium Chlorate-Nitric Acid Mixture—Dissolve 5 g of KClO<sub>3</sub> in 100 mL of HNO<sub>3</sub> (sp gr 1.42). Prepare this solution just before use and do not save any surplus for use later.

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

13.4 Sodium Hydroxide Solution (100 g/L)—Dissolve 100 g of NaOH in water and dilute to 1 L.

13.5 Sodium Thiosulfate, Standard Solution (0.0500 N)—Dissolve 12.4103 g of  $Na_2 S_2 O_3 \cdot 5H_2 O$  in water and dilute to 1 L in a volumetric flask.

13.6 Starch Indicator Solution—Make a paste of 1 g of soluble starch in about 5 mL of water, add 100 mL of water, and boil for 1 min while stirring. Cool and add 1 drop of chloroform. This solution is subject to decomposition and fresh solution shall be prepared if a dark-blue color is not produced with a drop of tincture of iodine in 100 mL of water on addition of a drop of the starch indicator solution.

# 14. Procedure for New Solutions

14.1 Transfer a portion of the sample containing the equivalent of about 0.11 g CuO to a 300-mL Erlenmeyer flask, and add 10 mL of water and 10 mL of HCl (sp gr 1.19). Wash down the side of the flask with water, boil for 1 min, and neutralize with NaOH solution until a permanent precipitate just forms. Add HCl (sp gr 1.19) dropwise until the precipitate just dissolves. Cool to below 20°C. The solution volume should be 20 to 30 mL.

14.2 Add 10 mL of KI solution and mix thoroughly by rotating the flask. Titrate with 0.05 N Na<sub>2</sub> S<sub>2</sub> O<sub>3</sub> solution, adding 2 mL of starch solution just before the brownish color of the iodine disappears. Stop the titration when the color changes from dark blue to cream color.

14.3 If poor end points or checks are obtained in the titrations (12.2), repeat the determination following the procedure described in Section 13.

# 15. Procedure for Used Solutions Contaminated with Organic Matter

15.1 In used solutions, the accumulation of organic matter may interfere with the copper analysis, resulting in inconsistent titrations in the determination of copper. In such cases, the organic matter may be destroyed as follows: Place the sample in a 300-mL Erlenmeyer flask, add 10 mL of the KClO $_3$ -HNO $_3$  mixture, and boil to dryness, with constant agitation ion. When dry, bake the residue over an open flame for about 1 min. Cool, then add 20 mL water and 10 mL of HCl (sp gr 1.19). Warm, if necessary, to effect complete solution.

15.2 Cool the solution and proceed in accordance with Section 14, starting with the second sentence of 15.1.

# 16. Calculation

16.1 Calculate the percentage of copper as CuO, as follows:

CuO, 
$$\% = (AB/C) \times 7.96$$
 (5)

where:

A =standard thiosulfate solution required for titration of the sample, mL,

B = normality of sodium thiosulfate solution, and

C = sample used, g.

#### **ZINC**

# 17. Reagents

17.1 Hydrochloric Acid, concentrated.

17.2 Hydrochloric Acid 1.20—Add one volume concentrated acid to 20 volumes water.

17.3 Alcohol, ethyl or isopropyl.

17.4 Hydrogen Sulfide Gas.

17.5 Ammonium Hydroxide, concentrated.

17.6 Uranyl Acetate, 1/10 N, 4.3 g dissolved in 100 mL water.

17.7 Potassium Ferrocyanide Solution , 0.15 N—Weigh accurately 21.12 g of pure  $K_4$  Fe(CN) $_6$ ·3H $_2$  O, and dissolve it in distilled water. Add 0.2 g sodium carbonate to stabilize the solution and make the volume up to 1.000 L. The solution shall be kept in a dark bottle. (See Sections 18 and 19.)

17.8 Zinc Chloride Standard Solution , 0.15 N—Weigh out 2.4518 g of pure zinc. Place the zinc in a 500 mL volumetric flask. Add 10 mL concentrated hydrochloric acid. After the initial reaction, dissolving may be hastened by heating. Cool to room temperature and dilute to mark. Use this solution for standardizing potassium ferrocyanide and back titrating procedure.

# 18. Procedure

18.1 Accurately weigh approximately 10 g of treating solution into a 500 mL Erlenmeyer flask. Add 5 mL concentrated HCI and 5 mL ethyl or isopropyl alcohol. Warm on hot plate and when alcohol fumes begin to come off, ignite them, leaving flask on the hot plate until the flame extinguishes. Dilute with 100 mL water and heat to boiling. Remove from hot plate and bubble  $\rm H_2$  S gas through until canary yellow precipitate comes after the initial black precipitate.

18.2 Filter—Use Whatman No. 40 paper receiving filtrate in 400 mL beaker. Wash precipitate with three 20 mL portions of 1:20 HCl. Boil solution down to about 50 mL to expel  $\rm H_2$  S. Cool. Add 75 mL water, 15 mL concentrated HCl, 12 mL concentrated NH<sub>4</sub> OH.

18.3 Titrate with  $K_4$  Fe(CN)<sub>6</sub> solution 0.15 N using uranyl acetate as an external indicator. Place two drops of indicator in each of a series of depressions of a glazed white ceramic spot plate or  $\frac{3}{4}$ -in. depressions in a block of paraffin. (The latter can best be made by cutting them out with the tip of a  $\frac{3}{8}$ -in. metal drill.)

18.4 If the approximate number of millilitres of K  $_4$  Fe(CN) $_6$  required is unknown, add 1 mL to the sample, stir thoroughly, then transfer a drop of the sample to one of the uranyl acetate spots. Wait 1 min. When the spot turns brown, the endpoint has been passed. Back titrate with  $\text{ZnCl}_2$  solution 0.15 N from a burette, adding a drop at a time, stirring thoroughly, then transferring a drop of the sample to the spot plate, and recording the reading of the burette after each drop is added. When the uranyl acetate spot remains colorless after a drop of sample is added, the endpoint has been passed in the opposite

direction. Match up the burette reading with the last drop in which the brown color is plainly visible.

18.5 Subtract the mL of  $ZnCl_2$  solution used to back titrate, from the mL of  $K_4$  Fe(CN)<sub>6</sub> and this will give the net mL of  $K_2$  Fe(CN)<sub>6</sub> required by the zinc in the sample.

# 19. Calculations

19.1 Calculate the percentage of active ingredient as follows:

$$Percentage = \frac{mL \ titration \times \ normality \ of \ K_4Fe(CN)_6 \times factor}{sample, \ g}$$
 (6)

Active Ingredi-	Factor
ents	
ZnO	4.068
ZnCl <sub>2</sub>	6.814
ZnSO <sub>4</sub>	14.373

Note 2—The procedure is based on the assumption that both the potassium ferrocyanide and zinc chloride solutions are made up to exactly 0.15 N. If some different weight (g) of zinc metal is used, calculate the normality of the zinc chloride for the procedure as follows:

Normality (ZnCl<sub>2</sub>) = 
$$\frac{\text{zinc, g} \times 2}{32.69}$$

The normality of potassium ferrocyanide is determined by pipetting a 25.00 mL aliquot of the standard zinc chloride solution into a 250 mL Erlenmeyer flask. Add 20 mL concentrated hydrochloric acid and 12.5 mL concentrated ammonium hydroxide, and then proceed in accordance with Steps 4, 5, 6, 7, and 8 of Standard A 2-59 (12).

Note 3—Normality of potassium ferrocyanide is calculated as follows:

Normality [K<sub>4</sub> Fe(CN)<sub>6</sub>] = 
$$\frac{\text{zinc, g}}{0.6538 \times \text{mL titration K}_4 \text{Fe(CN)}_6}$$

In the event that the normalities of the two solutions are not equal, a multiplier will have to be applied to the quantity of zinc chloride solution subtracted in step 5. Using the 25.00 mL zinc chloride and the potassium ferrocyanide titration volume obtained in the standardization, this multiplier (M) can be calculated:

$$M = \frac{K_4 \, Fe(CN)_6, \, mL}{ZnCl_2, \, mL}$$

Calculation in step 5:

net mL 
$$K_4$$
 Fe(CN)<sub>6</sub> = mL  $K_4$  Fe(CN)<sub>6</sub>—mL ZnCl<sub>2</sub> × M (7

# AMMONIUM CARBONATE

#### 20. Reagents

 $20.1\ CO_2\ Free\ Water$ —Distilled or demineralized water freshly boiled and stored in a closed container. Use whenever  $CO_2$  free water is called for.

20.2 Phenolphthalein, 1.0 % solution in 50 % alcohol.

20.3 Potassium Acid Phthalate, primary standard grade.

20.4 Sodium Hydroxide Solution, 0.1 N—Purchase standardized solution or dissolve 4.0 g sodium hydroxide in  $CO_2$ -free water, cool to room temperature and dilute to 1 L with  $CO_2$ -free water. (See Sections 21 and 22.)

20.5 Hydrochloric Acid Solution, 0.1 N—Purchase standardized solution or dilute 8.3 mL concentrated hydrochloric acid to 1 L with CO<sub>2</sub>-free water. (See Sections 21 and 22.)

20.6 CO<sub>2</sub> Absorbent, for drying tube.

20.7 Ammonium Hydroxide, reagent grade, 28 to 30 %.



20.8 Calcium Chloride Solution, 10 %, 100 g in 900 g CO<sub>2</sub>-free distilled water.

20.9 Sulfuric Acid Solution, 1:9, 10 g in 90 g water.

20.10 Apparatus:

20.10.1 Plastic Tubing Connector—Two piece.

20.10.2 Dropping Bottle.

20.10.3 Gas Washing Bottle.

20.10.4 Filter Flask-250 mL.

20.10.5 Magnetic Stirrer-Hot Plate.

20.10.6 Magnetic Stirring Bar, 1 in.

20.10.7 Aspirator (or Vacuum Pump).

20.10.8 Gas Drying Tube.

20.10.9 Glass Wool, for drying tube.

20.10.10 One-Hole Rubber Stoppers, #6 (to fit filter flask).

20.10.11 Plastic Thistle Tube, with removable funnel.

20.10.12 Vinyl or Latex Tubing, ¼ in. inside diameter.

# 21. Procedure

21.1 *Vacuum Flask* containing stirrer, one hole stopper, and thistle tube are set on stirrer-hot plate. The thistle tube shall extend as near bottom of flask as possible without interfering with stirring bar.

21.2 Add 200 mL water to gas bottle.

21.3 Connect vacuum flask tubule to the gas bottle tubule leading to the frittered glass gas disperser near the bottom of the bottle. Plastic two-piece tubing connectors on each side of the gas bottle make later manipulations quite convenient.

21.4 Connect the outlet of the gas bottle to the vacuum system, using a short section of light tubing with a pinch clamp on it next to the gas bottle.

21.5 Start vacuum system and adjust air to a moderate flow with pinch clamp.

21.6 Add about 50 to 60 mL water to thistle tube as it sucks into flask.

21.7 Using the pinch clamp, adjust airflow to about 2 to 4 bubbles per second through the bottom of the thistle tube.

21.8 Without disturbing the pinch clamp setting, disassemble and dump water.

21.9 Add 150 mL CO<sub>2</sub> free water and 50 mL reagent ammonium hydroxide to the gas bottle. Reassemble, check air flow for 2 to 4 bubbles per second and adjust if needed.

21.10 Weigh sample of solution containing 0.24 to 0.38 g of ammonium bicarbonate (or 0.13 to 0.21 g  $\rm CO_2$ ), into thistle tube. This is done conveniently with a weighed dropping bottle. Follow immediately with 50 mL  $\rm CO_2$ -free water. Note that 10 mL of a 6 % ACZA solution contains about 0.3 g of ammonium bicarbonate.

21.11 Turn on stirrer to medium stirring action, and heat high enough to boil in about 5 min.

21.12 As soon as heat switch is on, start adding 1:9 sulfuric acid about 1 mL at a time, until first sign of solids appear in flask. Then slow acid addition to a drop at a time, a drop to each 2 or 3 air bubbles. Rapid acid addition at this point will blow  $CO_2$  out the thistle tube. When solids have redissolved, add 2 mL excess 1:9 acid.

21.13 As soon as acid addition is complete (about 2 min), remove funnel top from thistle tube and attach drying tube filled with  $CO_2$  absorbent in place.

21.14 At first sign of boiling, or when heat reaches top of filter flask, shut heat off. Continue air flow 15 min more, to a total of about 20 min.

21.15 Disassemble, wash gas bottle contents into 500 mL wide mouth Erlenmeyer flask, using CO<sub>2</sub>-free water.

21.16 Add 10 mL 10 % calcium chloride solution, dilute to about 400 mL with  $\rm CO_2$ -free water, and digest on steam bath 2 h

21.17 Filter through asbestos mat in Gooch crucible, wash flask and crucible with  $\rm CO_2$ -free water. Return crucible to Erlenmeyer flask and dry in an oven at 225°F for 1 h.

21.18 Add 100 mL standardized hydrochloric acid (pipette). Tilt and rotate flask to wash sides and dissolve any adhering calcium carbonate. Dilute to about 150 mL with  $\rm CO_2$  free water, washing sides of flask.

21.19 Boil gently 1 min to expel CO<sub>2</sub>.

21.20 Titrate to phenolphthalein end-point with 0.1 *N* standardized sodium hydroxide.

21.21 Standardization of Sodium Hydroxide Solution—Weigh out replicate portions of potassium acid phthalate,  $1.6000~g~\pm~0.1000~g$ , transfer to Erlenmeyer flasks. Dissolve each in  $100~mL~CO_2$ -free water, adding 2 drops phenolphthalein. Titrate with the sodium hydroxide solution to a faint permanent pink color. Replicate titrations should yield normalities within 0.0005.

21.22 Standardization of Hydrochloric Acid Solution—Pipette 50 mL of hydrochloric acid solution into 250 mL Erlenmeyer flask. Add 2 drops phenolphthalein indicator and titrate with standardized sodium hydroxide solution until a faint permanent pink color appears. Duplicate titrations should agree within 0.1 mL. Record average.

21.23 Run blanks, that is, run the complete analysis without a sample added. At step 12, only the 2 mL excess of 1:9 sulfuric acid is needed. At step 18, use 50 mL of the standardized HCl as in step 22. The millilitres used to titrate this solution will be less than that used in the standardization, step 22. The difference is the blank used in the calculations.

#### 22. Calculations

22.1 Legend:

 $egin{array}{lll} N_{HCI} & = & Normality & HCl \\ N_{NaOH} & = & Normality & NaOH \\ AI & = & Active & Ingredient \\ F & = & Factor \\ \end{array}$ 

mL NaOH in step 22—mL NaOH in step 23 = blank

 $\frac{\text{grams potassium acid phthalate}}{\text{mL NaOH} \times 0.2042} = N_{\text{NaOH}} \tag{8}$ 

$$\frac{N_{\text{NaOH}} \times \text{mL NaOH}}{50} = N_{\text{HCI}}$$
 (9)

$$\frac{\left[\text{mL HCI} \times \text{N}_{\text{NaOH}} (\text{mL NaOH} + \text{Blank})\right] \times \text{F}}{\text{sample weight}} = \%AI \qquad (10)$$

Active Ingredient Factor
Ammonium bicarbonate 3.953
Carbon dioxide 2.201



# 23. Precision and Bias

# 24. Keywords

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

24.1 ammoniacal copper arsenate; ammoniacal copper zinc arsenate

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