Designation: D 3873 – 95

An American National Standard

Standard Test Method for
Valency State of the Arsenic Component of Ammoniacal Copper Arsenate Solutions[1]

This standard is issued under the fixed designation D 3873; 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 This test method is intended for testing batches of concentrated ammoniacal copper ammoniacal copper zinc arsenate (ACA/ACZA) solution, prepared by air-oxidation of trivalent arsenic. The method provides a means of confirming that oxidation has been completed and that at least 99.5 % of the arsenic has been oxidized. The sample for testing should contain 100 ± 20 mg of potential $As_2 O_5$. A200-mL aliquot of a concentrate containing 8 to 12 % of preservative oxides is suitable.

1.2 *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]

3. Summary of Test Method

3.1 Twenty millilitres of tartaric acid solution is added to a 250 mL Erlenmeyer flask, then 2 mL of the (ACA/ACZA) concentrate is added. The resulting solution should be light blue-green. Next, 20 mL of sodium bicarbonate solution is added. The solution will then be light blue. 2 ml of the starch indicator is added next. To this solution one drop of iodine solution from a buret is added. If the solution turns a dark blue and remains, it means the aeration has been completed.

4. Significance and Use

4.1 This method tests the completion of aeration which is used to convert trivalent arsenic to pentavalent arsenic.

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.[3] 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 as defined in Specification D 1193.

5.3 *Iodine Solution*—Add 6.3 and 6.4 g of resublimed iodine to a 500-mL standard flask. Add 10 to 12 g of potassium iodide and not more than about 20 mL of water. Swirl the contents of the flask at room temperature until all the iodine has dissolved, and dilute to the graduation mark. This solution can be kept for a few weeks if stored in a tight-sealed, glass-stoppered, dark bottle in a cool place.

5.4 *Sodium Bicarbonate Solution, Saturated*—Add 45 g of sodium bicarbonate to 400 mL of water, and swirl occasionally until most of the sodium bicarbonate goes into solution. Do not use heat to dissolve the salt. This solution can be kept in a stoppered flask.

5.5 *Starch Indicator Solution*—Make a paste of about 1.0 g of *soluble* starch in 5 mL of water. Pour the paste into 200 mL of water, near the boiling point, and then boil for 1 min. When the solution has cooled, add a few drops of chloroform as a preservative and keep in a stoppered bottle. Some batches of starch will not dissolve properly. If the solution separates into two layers, use the clear, top layer. If the whole solution is cloudy, prepare a fresh solution, or obtain a different supply of starch.

5.6 *Tartaric Acid Solution*—Dissolve 27 g of tartaric acid in 400 mL of water.

6. Procedure

6.1 Take a sample of the (ACA/ACZA) concentrate and place in a stoppered container (contact of the sample with air should be kept to a reasonable minimum or else misleading results could be obtained). Allow the sample to settle for at least 5 min before analysis.

6.2 Add 20 mL of the tartaric acid solution to a 250-mL Erlenmeyer flask.

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[1] This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

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

[3] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., New York, NY, and the "United States Pharmacopeia."

6.3 Using a rubber pipetting bulb, pipet exactly 2.00 mL of the (ACA/ACZA) concentrate into the tartaric acid solution. The color of the solution should now be light blue-green. If a reddish-gray, cloudy suspension is obtained, it means that aeration of the concentrate is required.

6.4 To the light blue-green solution, add 20 mL of saturated sodium bicarbonate solution. The solution will now be light blue. Add 2 mL of the starch indicator solution.

6.5 Add 1 drop of the iodine solution. This should be done from a buret so that the drop will be about 0.05 mL. Swirl the solution in the flask. If it immediately becomes dark blue (similar to the color of an (ACA/ACZA) treating solution), and remains dark for at least 1 min, it means that aeration of the concentrate was complete, and that at least 99.5 % of the arsenic is in the pentavalent form. If this result is not obtained, aeration should be continued, and another sample tested later.

7. Precision and Bias

7.1 Because this test method does not determine specific concentration, but is a drop pass/fail decision, a precision and bias statement does not apply.

8. Keywords

8.1 ACA; ACZA; arsenic; preservative; valency

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