Designation: D8496 – 23

Standard Test Method for Determination of Hydroquinone (HQ) Content in Vinyl Acetate Monomer (VAM) Using Ultraviolet-Visible (UV-Vis) Spectrophotometer[1,2]

This standard is issued under the fixed designation D8496; 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 covers the determination of hydroquinone (HQ) in colorless vinyl acetate monomer (VAM). This test method is applicable to the determination of HQ in the concentration range from 1 to 25 ppm.

1.2 For purposes of determining conformance of an observed or a calculated value using this test method to relevant specifications, test result(s) shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit in accordance with Practice E29.

1.3 For ensuring safety, hazard information and guidance, follow the manufacturer's material safety data sheet.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1152 Specification for Methanol (Methyl Alcohol) (Withdrawn 2021)[4]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Summary of Test Method

3.1 This test method is quick, and it is easier to measure the amount/quantity of hydroquinone (HQ) in vinyl acetate monomer (VAM) quantitatively by using an ultraviolet-visible (UV-Vis) spectrophotometer following the Beer-Lambert Law[5] principle at absorbance of 293 nm and methanol as a blank.

3.2 In this process, neither color development/reaction nor special reagent/processing is required, and the VAM sample can be analyzed directly without further processing.

4. Significance and Use

4.1 VAM is an organic vinyl ester compound. This colorless liquid is the precursor to polyvinyl acetate, an important industrial polymer. VAM may be subject to rapid spontaneous polymerization if the inhibitor is not present or becomes depleted during prolonged storage.

4.2 VAM is typically shipped with a HQ inhibitor as free radical scavenger. VAM should be evaluated to ensure that appropriate systems (for example, temperature and inhibitor quantity) are assured during storage, transportation, and getting prolonged shelf life.

4.3 Most VAM shipped from the manufacturer will contain an inhibitor, typically 3 to 5 ppm HQ for regional shipments and up to 25 ppm HQ for long-range shipments.

5. Interferences

5.1 Methanol and VAM have no interference at 293 nm in a UV-Vis spectrophotometer.

6. Apparatus

6.1 *UV-Vis Spectrophotometer*, absorbance at 293 nm.

6.2 *Volumetric Flask*, 50 mL and 100 mL capacity.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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[2] Provided by the Berger Paints Bangladesh Limited, Berger House, Rd No 2, Uttara, Dhaka 1230, Bangladesh.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] Clark, Jim. "The Beer-Lambert: Law Chemistry," *LibreTexts*.

6.3 *Pipets or Auto-Pipets*, 1 mL or 2 mL capacity.

6.4 *Quartz Cuvette*, material: Q, path Length: 10 mm, and Match Code: 6.

6.5 *Beakers*, 50 mL and 250 mL capacity.

7. Reagents and Materials

7.1 *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.[6] Three 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.

7.2 *Purity of Methanol (Purity—Minimum 99 %)*—Unless otherwise indicated, references to water shall be understood to mean methanol conforming to Specification D1152.

7.3 *HQ (Purity—Minimum 99 %)*—Reagent-grade chemicals shall be used.

8. Hazards

8.1 Store samples of VAMs in amber bottles or protect from light by other means to aid in preventing polymerization. Keep samples away from heat sources and chemicals that can cause free radical polymerization. VAM can polymerize violently, evolving considerable heat.

9. Preparation of Apparatus

9.1 Switch on the UV-Vis spectrophotometer ½ h before taking measurements.

10. Calibration and Standardization

10.1 Weigh 0.10 g of HQ to the nearest 0.1 mg into a 100 mL volumetric flask containing approximately 50 mL of methanol. Mix well until the solution is completely dissolved. Dilute the solution with methanol up to the mark. This is the stock solution of HQ (1000 ppm).

10.2 Prepare a series of standards by pipetting 0.25 mL, 0.50 mL, 0.75 mL, 1.00 mL, and 1.25 mL portions of the HQ solution into respective 50 mL volumetric flasks. Dilute each flask to the mark with methanol and mix well. These standards contain approximately 5 ppm, 10 ppm, 15 ppm, 20 ppm, and 25 ppm of HQ, respectively.

10.3 Determine the absorbance of each of these standards at 293 nm using methanol as the blank with the UV-Vis spectrophotometer.

10.4 Construct a calibration curve on rectangular coordinate graph paper or software of UV-Vis spectrophotometer by plotting the absorbance of the calibration standards at 293 nm.

11. Procedure

11.1 No sample preparation is required, and VAM can be measured directly. Using the procedure followed for the calibration, determine the absorbance of the solution at 293 nm using methanol as the blank with the UV-Vis spectrophotometer.

11.2 From the calibration curve, determine the HQ content corresponding to the absorbance obtained.

12. Calculation or Interpretation of Results

12.1 Calculate the HQ content corresponding to the absorbance (y) obtained from the calibration curve by using software of the UV-Vis spectrophotometer or a regression line equation of calibration curve:

$$(y = ax + c),\ x = (y - c)/a \tag{1}$$

where:

y = absorbance of sample,
a = slope of regression line of calibration,
c = intersect of regression line of calibration, and
x = HQ quantity.

13. Report

13.1 Report the concentration of HQ in ppm.

14. Precision and Bias

14.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same analyst should be considered suspect if they differ by more than 0.5 ppm.

14.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by analysts in different laboratories should be considered suspect if they differ by more than 0.8 ppm.

NOTE 1—The preceding precision statements are based upon an interlaboratory study on one sample of vinyl acetate containing 15.3 ppm HQ. Each sample was analyzed in ten number replicates on two different days by one analyst in each of two different laboratories.

14.2 *Bias*—This bias of this test method has not been determined because there is no appropriate standard available.

15. Keywords

15.1 Beer-Lambert Law; hydroquinone; inhibitor; UV absorbance; vinyl acetate monomer

[6] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.