Designation: D2193 – 22

Standard Test Method for Hydroquinone in Vinyl Acetate[1]

This standard is issued under the fixed designation D2193; 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 in the range from 1 to 20 ppm in refined, commercially available, vinyl acetate.

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

1.4 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

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:*[2]

D1193 Specification for Reagent Water

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

3. Summary of Test Method

3.1 The vinyl acetate is evaporated at room temperature in a stream of inert gas or clean air to minimize the loss of hydroquinone by evaporation. The hydroquinone is dissolved in water and titrated with dilute standardized ceric acid sulfate using diphenylamine as indicator.

4. Significance and Use

4.1 This test method provides a measurement of inhibitor level in vinyl acetate. The results of these measurements can be used for specification acceptance.

5. Apparatus

5.1 *Buret,* 25-mL, graduated in 0.1-mL subdivisions.

5.2 *Beakers,* 50 and 600-mL capacity.

5.3 *Volumetric Flask,* 1000-mL capacity.

5.4 *Erlenmeyer Flasks,* 100 and 250-mL capacity.

5.5 *Nitrogen Cylinder,* or source of clean air.

5.6 *Pipets,* 10 and 50-mL capacity.

6. Reagents

6.1 *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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

6.3 *Ceric Acid Sulfate, Standard Solution (0.002 N)*—Dissolve 1.096 g of ceric ammonium nitrate ($(NH_4)_2Ce(NO_3)_6$) in 28.0 mL of concentrated sulfuric acid (H_2SO_4, sp

[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] 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.

[3] *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.

gr 1.84) contained in a 50-mL beaker. Slowly pour the ceric solution, while stirring, into 200 mL of water contained in a 600-mL beaker. When solution is complete, transfer this mixture to a 1000-mL volumetric flask and dilute to the mark with water.

6.4 *Diphenylamine Indicator Solution*—Dissolve 0.1 g of diphenylamine in 100 mL of H_2SO_4 (sp gr 1.84) and store this solution in a brown glass bottle.

6.5 *Hydroquinone Standard*—Dissolve 200.0 mg of hydroquinone, weighed to the nearest 0.1 mg, in water and dilute to 1000.0 mL in a volumetric flask. This solution is unstable and should be discarded after 1 week of normal use.

7. Standardization

7.1 Pipet 10-mL portions of the hydroquinone standard (see 6.5) into each of two 100-mL Erlenmeyer flasks. Add 3 drops of diphenylamine indicator solution to each flask. Using a 25-mL buret, titrate the contents of each flask with ceric acid sulfate solution to a faint blue end point that is permanent for 15 s. The titrations should be approximately 20 mL and should agree within 0.5 mL. Average the two values and use in the calculations (Section 9).

8. Procedure

8.1 Pipet 50 mL of the vinyl acetate sample into each of two 250-mL flasks.

8.2 Evaporate the specimens at room temperature by passing a stream of cylinder nitrogen gas or clean air into the flasks. Bench-line air should be passed through a fiberglass filter before entering the specimen flasks. Maintain the flow of gas just short of a level causing splattering of the specimen. That part of the delivery tube in the flask must be of metal, glass, or an inert plastic, such as polyethylene or polytetrafluoroethylene (PTFE).

8.3 After complete evaporation, which requires 45 to 60 min, remove the gas stream and dissolve the hydroquinone in 25 mL of water.

8.4 Add 3 drops of diphenylamine indicator solution to each flask using the same dropper as in the reagent standardization. Titrate each solution with the ceric acid sulfate reagent to a light blue end point that is permanent for 15 s.

9. Calculation

9.1 Calculate the parts per million of hydroquinone, *H*, in the sample as follows:

$$H = \left[(V \times F)/S \right] \times 1000 \quad (1)$$

where:

V = millilitres of ceric acid sulfate reagent required for titration of the specimen, (see 8.4),
F = factor (Section 7) = milligrams of hydroquinone in 10-mL aliquot/average millilitres of ceric acid sulfate reagent, and
S = grams of sample used = 50 × specific gravity.

10. Report

10.1 Report the concentration of hydroquinone to the nearest 0.1 ppm.

11. Precision and Bias

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

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same analyst should be considered suspect if they differ by more than 0.3 ppm.

11.1.2 *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 1.0 ppm.

NOTE 1—The preceding precision statements are based upon an interlaboratory study on two samples of vinyl acetate containing 4.6 and 15.3 ppm hydroquinone. Each sample was analyzed in duplicate on two different days by one analyst in each of five different laboratories.

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

12. Keywords

12.1 HQ; hydroquinone; vinyl acetate