Designation: D3125 – 06 (Reapproved 2012)

# Standard Test Method for Monomethyl Ether of Hydroquinone in Colorless Monomeric Acrylate Esters and Acrylic Acid[1]

This standard is issued under the fixed designation D3125; 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 monomethyl ether of hydroquinone[2] (MEHQ) in colorless monomeric acrylate esters and acrylic acid. The test method is applicable to the determination of MEHQ in the concentration range from 0 to 1200 parts per million.

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 the rounding-off method of 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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

## 2. Referenced Documents

2.1 *ASTM Standards:*[3]

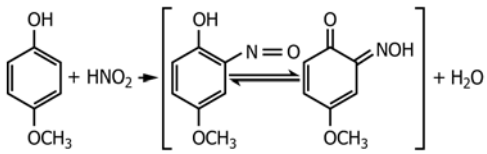
D1193 Specification for Reagent Water

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

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[4]

## 3. Summary of Test Method

3.1 As shown in the equation, MEHQ reacts with nitrous acid (sodium nitrite in acidic media) to form the nitroso derivative which equilibrates between two structures.




3.2 The yellow color of the nitroso compound is measured spectrophotometrically at a wavelength of 420 nm.

## 4. Significance and Use

4.1 Acrylic acid and its esters are normally inhibited with MEHQ only. This procedure presents a rapid and accurate method of determining the MEHQ content of fresh acrylic acid and acrylate esters in the absence of other inhibitors.

4.2 MEHQ effectiveness may decline with age and this decline in effectiveness may not be indicated by this test method.

## 5. Interferences

5.1 Hydroquinone (HQ), thiodiphenylamine, diphenylphenylene-diamine and *p*-hydroxydiphenylamine interfere if present.

## 6. Apparatus

6.1 *Spectrophotometer,* with borosilicate-glass cells for determining absorbance at 420 nm.

6.2 *Volumetric Flasks,* 50 and 100-mL capacity.

6.3 *Measuring Pipets,* 5 and 10-mL capacity.

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, it is intended that all reagents

[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] IUPAC-approved name is 4-methoxyphenol.

[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.

***A Summary of Changes section appears at the end of this standard**

**TABLE 1 Amount of Specimen**

| Expected MEHQ Content, ppm | Amount of Specimen, g |
|---|---|
| 0 to 25 | 25[A] |
| 25 to 100 | 10[A] |
| 100 to 250 | 5[B] |
| 250 to 550 | 2[B] |
| 550 to 1000 | 1[B] |

[A] Weigh to the nearest 10 mg.
[B] Weigh to the nearest 1 mg.

**TABLE 2 Reporting and Averaging of Duplicate Runs**

| MEHQ Concentration, ppm | Report ppm | Duplicate Runs That Agree Within the Following Amounts Are Suitable for Averaging (95 % Confidence Level) |
|---|---|---|
| | | ppm |
| 15 | 0.1 | 0.54 |
| 50 | 0.5 | 1.8 |
| 200 | 1 | 5.5 |
| 500 | 1 | 9.9 |

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.

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

7.3 *Acetic Acid,* glacial.

7.4 *Monomethyl Ether of Hydroquinone (MEHQ)* (4-methoxyphenol).

7.5 *Sodium Nitrite Solution (2 %)*—Dissolve 2 g of sodium nitrite ($NaNO_2$) in water and dilute to 100 mL.

## 8. Hazards

8.1 Store samples of acrylic monomers 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. Acrylic monomers can polymerize violently, evolving considerable heat. Keep sample container size to a minimum. The inhibitor, monomethyl/ester of hydroquinone, requires oxygen to remain active.

## 9. Calibration

9.1 Weigh 0.10 g of MEHQ to the nearest 0.1 mg into a 100-mL volumetric flask containing approximately 50 mL of glacial acetic acid. Mix well until solution is complete then dilute to the mark with glacial acetic acid. Prepare a series of standards by pipetting 1, 2, 4, 6, and 10-mL portions of the MEHQ solution into respective 50-mL volumetric flasks. Dilute each flask to the mark with glacial acetic acid and mix well. A10-mL aliquot of each of these standards contains approximately 200, 400, 800, 1200, and 2000 µg of MEHQ, respectively.

9.2 Determine the absorbance of each of these standards by pipetting 10-mL aliquots into 50-mL volumetric flasks containing 20-mL of glacial acetic acid. To each flask add 1-mL of 2 % $NaNO_2$ solution and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min. With cells appropriate to the instrument, determine the absorbance at 420 nm using acetic acid as the blank.

9.3 Construct a calibration curve on rectangular coordinate graph paper by plotting the absorbances of the standards at 420 nm against the micrograms of MEHQ.

## 10. Procedure

10.1 Perform analyses in duplicate and carry a blank through the analysis using 49 mL of glacial acetic acid in place of the specimen solution.

10.2 Weigh the appropriate amount of specimen (Table 1) into a 50-mL volumetric flask containing 20-mL of glacial acetic acid.

10.3 Add 1 mL of 2 % $NaNO_2$ solution to the specimen and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min.

10.4 Using the procedure followed for the calibration, determine the absorbance of the solution at 420 nm with the blank solution in the reference position. From the calibration curve, determine the micrograms of MEHQ corresponding to the absorbance obtained.

## 11. Calculation

11.1 Calculate the concentration of MEHQ in ppm as follows:

$$MEHQ, \text{ ppm} = M/S \quad (1)$$

where:

$M$ = micrograms of MEHQ from calibration curve and
$S$ = grams of specimen used in the test.

## 12. Report

12.1 Report the concentration of MEHQ as indicated in Table 2.

## 13. Precision[6]

13.1 The precision statements are based upon an interlaboratory study in which one operator in each of twelve laboratories analyzed in duplicate on two different days each of the following samples:

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1016. Contact ASTM Customer Service at service@astm.org.

| Acrylic Monomer | Mean MEHQ Concentration, ppm |
|---|---|
| Ethyl acrylate | 16.9 |
| Ethyl acrylate | 48.2 |
| 2-Ethylhexyl acrylate | 14.7 |
| 2-Ethylhexyl acrylate | 47.3 |
| Acrylic acid | 212 |
| Acrylic acid | 499 |

13.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 4.0 % relative.

13.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 15 % relative.

13.2 *Bias*—Bias cannot be determined for this test method because there is no available material having an accepted reference value.

## 14. Keywords

14.1 acrylate esters; acrylic acid; monomeric ester of hydroquinone

## SUMMARY OF CHANGES

Committee D01.35 has identified the location of selected changes to this standard since the last issue (D3125 - 97 (2001)) that may impact the use of this standard. (Approved June 1, 2006.)

*(1)* Added reference to Practice E29 in the Scope section.

*(2)* Added Practice E29 to list of Referenced Documents.