



Designation: E346 – 24

Standard Test Method for Analysis of Carbonizables in Methanol¹

This standard is issued under the fixed designation E346; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of carbonizables in methanol.

1.2 This test method is applicable to samples with carbonizables concentrations to 60 Pt-Co units.

1.3 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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.* Specific hazards statements are given in Sections 9.

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

D5386 Test Method for Color of Liquids Using Tristimulus Colorimetry

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

D8005 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

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

E300 Practice for Sampling Industrial Chemicals

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1547 Terminology Relating to Industrial and Specialty Chemicals

2.2 *Other Document:*³

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200 – Air Contaminates – Table of Exposure Limits and Hazard Communication

3. Terminology

3.1 See Terminology E1547 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 Methanol is mixed with a known volume of concentrated sulfuric acid under controlled conditions. The color formed by the action of the acid on the carbonizable impurities in the methanol is estimated by comparison of the test mixture with platinum-cobalt color standards.

5. Significance and Use

5.1 This test method is suitable for manufacturing control and for determining compliance with specification limits.

6. Interferences

6.1 Because this test method is designed to measure low concentrations of impurities that carbonize or darken in the presence of concentrated sulfuric acid, erroneously high results may be obtained if all glassware is not clean.

¹ These test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.14 on Alcohols & Glycols.

Current edition approved May 1, 2024. Published June 2024. Originally approved in 1968 as E346 – 68 T. Last previous edition approved in 2008 as E346 – 08^{ε1} which was withdrawn March 2017 and reinstated in May 2024. DOI: 10.1520/E0346-24.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

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

7. Apparatus

7.1 *Erlenmeyer Flask*, 125 mL borosilicate glass.

7.2 *Nessler Tubes*, 50 mL high form, matched.

7.3 *Ring Stand*.

7.4 *Buret*, 25 mL, with TFE-fluorocarbon stopcock.

NOTE 1—A 25 mL automatic buret graduated in 0.1 mL increments provides a safe convenient way of dispensing the sulfuric acid and protects the acid from dust and other contamination.

7.5 *Electric Stirrer and Bar*.

8. Reagents

8.1 *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.⁴ 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.

8.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification **D1193**.

8.3 *Sulfuric Acid*—Concentrated sulfuric acid (sp gr 1.84) CAS No.7664-93-9.

8.4 *Platinum-Cobalt Stock Solution and Color Standards*, made in accordance with Test Method **D5386** or **D8005**.

9. Hazards

9.1 **Warning**—Methanol is toxic both as a liquid and as a vapor, and is dangerous if not properly handled. Any person sampling or handling this product should have consulted a Safety Data Sheet (SDS) for specific first aid instructions and information on the proper equipment to have available for use in the event of personal contact or exposure.

9.2 **Warning**—Concentrated sulfuric acid is corrosive; contact with the body is to be avoided at all times. Use proper protective equipment, including adequate eye protection. If the eyes are affected or if a burn results, immediately rinse eyes with copious amounts of water for 15 min to 20 min. Then obtain immediate medical attention.

⁴ 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.

10. Sampling

10.1 For details of sampling methanol, refer to Practice **E300**.

11. Procedure

11.1 All glass apparatus used for this test method must be kept free of materials which produce color with sulfuric acid. Clean all glassware with an effective detergent or other cleaning agent that effectively cleans the glassware. Rinse with water. Water will sheet on glassware if it is clean. Dry with clean air or rinse with methanol that is known to give little or no color with sulfuric acid. (**Warning**—Do not use acetone to dry apparatus.)

11.2 Transfer 50 mL of the proper platinum-cobalt color standard into one of the matched 50 mL Nessler tubes.

11.3 Pipet 30 mL of the sample into a 125 mL Erlenmeyer flask.

11.4 Add, at a uniform rate, 25 mL of H₂SO₄ to the sample while stirring constantly using an electric stirrer and stirring bar. The total time of the acid addition shall be 5 min ± 30 s. (**Warning**—Do not cool the mixture.)

11.5 Allow the mixture to stand for 15 min ± 30 s at room temperature, pour the mixture from the flask into a 50 mL Nessler tube and compare the color of the sample to the proper platinum-cobalt standard by looking down through the longitudinal axis of the tubes upon a white or mirrored surface at such an angle that light is reflected through the column of liquid. Hold the tubes at some convenient height 50 mm to 150 mm from the surface.

12. Report

12.1 Report the Pt-Co unit to the nearest 1 for samples with Pt-Co unit values up to 20 Pt-Co units. For results above 20 Pt-Co units, report the estimated units between the two standards.

13. Precision and Bias

13.1 *Precision*—The following criteria should be used for judging the acceptability of results (**Note 2**).

13.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 1.7 units at 21 df. The 95 % limit for the difference between two such runs is 5 units.

13.1.2 *Laboratory Precision (Within-Lab Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the value in **Table 1** at the indicated

TABLE 1 Carbonizables Precision Values, Pt-Co Units

Pt-Co Level	Laboratory Precision Within-Lab, Between-Days			Reproducibility		
	Standard Deviation	Degrees of Freedom	95 % Limit	Standard Deviation	Degrees of Freedom	95 % Limit
5	1	13	3	2	6	5
15	1	13	3	3	6	10
60	1	13	3	5	6	15