Designation: D2194 – 02 (Reapproved 2012)

Standard Test Method for Concentration of Formaldehyde Solutions[1]

This standard is issued under the fixed designation D2194; 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 Department of Defense.

1. Scope*

1.1 This test method covers the determination of the formaldehyde content of commercially available formaldehyde solutions ranging in concentration from 36 to 55 weight %.

1.2 For purposes of determining conformance of an observed value 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 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:*[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 specimen is reacted with an excess of sodium sulfite solution and the resulting sodium hydroxide is titrated with sulfuric acid using thymolphthalein indicator.

3.2 The sample should be essentially neutral; 0.1 % acidity (as formic acid) is equivalent to 0.065 % formaldehyde.

4. Significance and Use

4.1 This test method provides a measurement of formaldehyde content (assay) of formaldehyde solutions. The results of these measurements can be used for specification acceptance.

5. Apparatus

5.1 *Buret,* calibrated, 100-mL, with a 50 or 75-mL reservoir on top of a lower portion calibrated in 0.1-mL divisions. A TFE-fluorocarbon resin stopcock is suitable for this purpose.

5.2 *Erlenmeyer Flask,* 500-mL capacity.

5.3 *Vials,* specimen, short, style, 1 to 1½-dram (4 to 6-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 *Sodium Sulfite Solution* (125 g/L)—Dissolve 125 g of anhydrous sodium sulfite (Na_2SO_3) in water and dilute to 1 L.

NOTE 1—Sodium sulfite gradually oxidizes to sodium sulfate on exposure to air and therefore should be kept in a tightly closed container. For best results freshly prepared reagent should be used.

6.4 *Sulfuric Acid* (0.5 N)—Prepare and standardize 0.5 *N* sulfuric acid (H_2SO_4) against 0.5 *N* sodium hydroxide (NaOH)

[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] *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.

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

solution which has been standardized against potassium acid phthalate to a thymolphthalein end point.

6.5 *Thymolphthalein Indicator* (0.1 % Alcohol Solution)—Dissolve 1.0 g of thymolphthalein in 100 mL of methanol, ethanol, or isopropanol and dilute to 1 L with additional alcohol.

7. Procedure

7.1 Add 100 mL of Na_2SO_3 solution to a sufficient number of 500-mL Erlenmeyer flasks to make all blank and specimen determinations in duplicate.

7.2 Weigh a 2-g specimen to the nearest 0.1 mg in the weighing vials and transfer the vials to the Erlenmeyer flasks, being careful to avoid getting any of the sample on the sides of the flasks.

7.3 Add 3 to 5 drops of the thymolphthalein indicator solution to each of the flasks containing specimens as well as the blanks and titrate to a colorless end point.

8. Calculation

8.1 Calculate the weight percent of formaldehyde, *W*, as follows:

$$W = [(V - B) \times N \times F]/S \times 100 \quad (1)$$

where:

V = H_2SO_4 required for titration of the specimen, mL,
B = H_2SO_4 required for titration of the blank, average, mL,
N = normality of the H_2SO_4,
F = 0.03003 (the milliequivalent weight of formaldehyde), and
S = sample used, g.

9. Report

9.1 Report the percent formaldehyde to the nearest 0.01 %. Duplicate runs that agree within 0.12 % are acceptable for averaging (95 % confidence level).

10. Precision and Bias

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

10.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations, obtained by the same analyst on different days is normally about 0.05 % absolute. Two such results should be considered suspect if they differ by more than 0.12 % absolute.

10.1.2 *Reproducibility*—The average difference between two results (each the mean of duplicate determinations) obtained by analysts in different laboratories is normally about 0.08 % absolute. Two such results should be considered suspect if they differ by more than 0.23 % absolute.

NOTE 2—The above precision estimates are based on two interlaboratory studies involving five and nine laboratories, respectively, using two different samples in each case with a single analyst performing duplicate results on each of two days. The formaldehyde levels studied were as follows: 36.85 %, 36.98 %, 37.15 %, and 37.23 %.

10.2 *Bias*—Bias has not been determined for this test method because primary standards do not exist.

11. Keywords

11.1 concentration; formaldehyde

SUMMARY OF CHANGES

Committee D01.35 has identified the location of selected changes to this standard since the last issue (D2194 - 99) that may impact the use of this standard.

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

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