Designation: D2119 – 09

Standard Test Method for Aldehydes in Styrene Monomer[1]

This standard is issued under the fixed designation D2119; 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 wet chemical determination of aldehydes in styrene monomer. Aldehydes are calculated and reported as benzaldehyde. The range of concentration for this test method is 0.001 % to 0.030 %.

1.2 In determining conformance of the test results using this method to applicable specifications, results shall be rounded off 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 *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.* For specific hazard statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D3437 Practice for Sampling and Handling Liquid Cyclic Products

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

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

2.2 *Other Documents:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and1910.1200[3]

3. Summary of Test Method

3.1 An alcoholic solution of hydroxylamine hydrochloride is added to a specimen of styrene monomer. Active aldehydes present react in accordance with the following equation:

$$RCHO + NH_2OH \cdot HCl \rightarrow RCHNOH + H_2O + HCl$$

The hydrochloric acid, which is equivalent to the aldehyde present in the sample, is titrated with standard sodium hydroxide solution.

4. Significance and Use

4.1 This test method is suitable for determining the quantity of aldehydes, both for quality control and quality assurance of the product.

5. Interferences

5.1 Ketones, if present, interfere by partially reacting with the reagent.

6. Apparatus

6.1 *Erlenmeyer Flasks,* glass-stoppered, 250-mL.

6.2 *Pipets,* 25-mL.

6.3 *Volumetric Flasks,* 100-mL.

6.4 *Burets,* 10-mL. (Microburets are preferred.)

6.5 *Thermometers,* capable of differentiating 1°C at ambient.

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 conform to the specifications of the Committee on

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

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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] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://www.dodssp.daps.mil.

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

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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D1193.

7.3 *Hydrochloric Acid, Standard (0.05 N)*—Prepare a 0.05 *N* solution of hydrochloric acid (HCl) by diluting 4.15 mL of concentrated HCl (density 1.19 g/mL) to 1 L with water.

7.4 *Hydroxylamine Hydrochloride Solution*—Dissolve 20 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) in 1 L of methanol and neutralize to the red-yellow end point of thymol blue indicator.

7.5 *Methanol.*

7.6 *Sodium Hydroxide, Standard Solution (0.05 N)*—Dissolve 2.00 g of low-carbonate sodium hydroxide (NaOH) in water and dilute to 1 L. Standardize against primary standard benzoic acid.

7.7 *Thymol Blue Indicator Solution*— Dissolve 0.1 g of thymol sulfonphthalein, sodium salt, in water and dilute to 100 mL.

8. Hazards

8.1 Consult the latest OSHA regulations, supplier's Material Safety Data Sheets, and local regulations regarding all materials used in this test method.

8.2 Styrene monomer is flammable and polymerizes exothermally on contact with peroxides, mineral acids, and $AlCl_3$.

9. Sampling and Handling

9.1 Collect the sample as directed in Practice D3437.

10. Procedure

10.1 Pipet 25.0 mL of the sample (**Warning—**see 8.2) into a 250-mL glass-stoppered Erlenmeyer flask containing 25 mL of methanol. Record the temperature of the sample. Add 0.2 mL of thymol blue indicator and, if necessary, neutralize with 0.05 *N* NaOH solution or 0.05 *N* HCl to the red-yellow end point (do not record). Add 25 mL of the neutralized hydroxylamine hydrochloride solution and allow to stand 1 h, shaking the flask occasionally. Titrate to the original red-yellow end point with 0.05 *N* NaOH solution, dispensed from a 10-mL buret, and record the volume used. Let stand 1 h and again titrate any acid that may have been liberated. Record the total volume of NaOH used.

10.2 Since methanol may contain aldehydes, run a blank determination on 25 mL of methanol.

11. Calculation

11.1 Calculate the percentage of aldehydes as benzaldehyde as follows:

$$\text{Aldehydes, \%} = [((A - B)N \times 0.106)/25C] \times 100$$

where:

A = NaOH solution required for titration of the specimen, mL,
B = NaOH solution required for titration of the methanol blank, mL,
N = normality of NaOH solution used, and
C = density of styrene monomer.

12. Report

12.1 Report the aldehydes content to the nearest 0.001 %.

13. Precision and Bias

13.1 *Intermediate Precision (formerly called Repeatability)*—Duplicate results by the same operator should not be considered suspect (95 % confidence limit) unless they differ by more than the following:

Aldehyde Content, %	Repeatability, %
0.004	0.0006

13.2 *Reproducibility*—The averages of duplicate results submitted by each of two laboratories should not be considered suspect (95 % confidence limit) unless they differ by more than the following:

Aldehyde Content, %	Reproducibility, %
0.004	0.0016

13.3 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method for measuring aldehydes, bias has not been determined.

14. Quality Guidelines

14.1 Refer to Guide D6809 for suggested QA/QC activities that can be used as part of this test method. It is recommended that the operator of this test method select and perform relevant QA/QC activities like the ones in Guide D6809 to help ensure the quality of the data generated by this method.

15. Keywords

15.1 aldehyde content; aldehydes in styrene; styrene

[4] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D2119 - 03) that may impact the use of this standard. (Approved June 1, 2009.)

(1) Changes made to conform to D16 Editorial Guidelines in Sections 1, 2, 7, and 9 as well as added Section 14.