Designation: D 2340 – 03

Standard Test Method for Peroxides in Styrene Monomer[1]

This standard is issued under the fixed designation D 2340; 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 the peroxide content of styrene monomer.

1.2 In determining the 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 E 29.

1.3 *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 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

2.2 *Other Documents:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[5]

3. Summary of Test Method

3.1 A specimen of styrene monomer is added to a solution of isopropanol and acetic acid. A saturated solution of sodium iodide in isopropanol is added and the solution refluxed. The peroxides present liberate iodine from sodium iodide quantitatively. The liberated iodine is then titrated with sodium thiosulfate to a colorless end point.

4. Significance and Use

4.1 This test method is suitable for determining the quantity of peroxides in styrene monomer both for quality control and quality assurance of the product.

5. Apparatus

5.1 *Erlenmeyer Flasks*, glass-stoppered, 500-mL, equipped with 300-mm Liebig condensers having inner and outer standard taper joints.

5.2 *Electric Hot Plate* with totally enclosed heating unit.

5.3 *Boiling Chips*.

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.[6] 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 as defined by Type III of Specification D 1193.

6.3 *Glacial Acetic Acid*.

6.4 *Isopropyl Alcohol*.

6.5 *Sodium Iodide Isopropyl Alcohol Solution*—Prepare a saturated solution of sodium iodide in isopropanol (approximately 200 g NaI/L).

6.6 *Sodium Thiosulfate, Standard Solution* (0.01 *N*)—Dissolve 2.5 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) and 0.1

[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 C_9 and C_{10} Aromatic Hydrocarbons.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 06.04.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

[6] *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.**

g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L. Standardize against primary standard potassium dichromate ($K_2Cr_2O_7$).

7. Hazards

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

7.2 Styrene monomer is flammable and polymerizes exothermally on contact with peroxides, mineral acids, and aluminum chloride.

7.3 Isopropyl alcohol is flammable and should be kept away from open flame and spark-producing apparatus. Use only a hot plate with totally enclosed heating unit in this analysis.

8. Sampling

8.1 Collect the sample as directed in Practice D 3437.

9. Procedure

9.1 Add 200 mL of isopropyl alcohol into each of two 500-mL Erlenmeyer flasks containing several boiling chips. Add 10 mL of glacial acetic acid to each flask. Into one flask pipet 50 mL of the styrene monomer sample. Identify this flask as "Sample" and the other flask as" Blank." Fit the condenser in place (**Warning:** see 7.2 and 7.3). Heat the contents of the flasks to boiling and pipet 50 mL of the saturated NaI isopropyl alcohol solution into each.

9.2 Continue boiling gently for 10 min. At the end of the boiling period, remove the flasks from the heat source. Rinse each condenser with two 10-mL portions of water, adding the rinsings to the respective flasks. Cool the flasks to room temperature. Titrate the liberated iodine in each flask with 0.01 *N* $Na_2S_2O_3$ solution to a light yellow color and continue to titrate slowly until the yellow color just disappears.

10. Calculation

10.1 Calculate the peroxide content of the specimens as hydrogen peroxide, in parts per million (mg/kg) as follows:

$$\text{Peroxides, mg/kg} = [(A - B) \times N \times 1.7 \times 10^4] / (50 \times C)$$

where:

- A = total millilitres of $Na_2S_2O_3$ solution required for titration of the specimen,
- B = total millilitres of $Na_2S_2O_3$ solution required for titration of the blank,
- N = normality of $Na_2S_2O_3$ solution used, and
- C = density of styrene monomer at temperature pipetted (an approximate density of 0.9 may be used to determine the sample weight).

11. Report

11.1 Report the peroxide content to the nearest 1 mg/kg.

12. Precision and Bias

12.1 *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:

Peroxide Content, mg/kg	Repeatability, mg/kg
1 to 60	6

12.2 *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:

Peroxide Content, mg/kg	Reproducibility, mg/kg
1 to 60	13

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

13. Keywords

13.1 peroxide content; peroxide in styrene; styrene

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D 2340 - 96) that may impact the use of this standard.

(1) This standard was editorially revised in 2003 to include a Bias statement.