



Designation: E 299 – 97

Standard Test Method for Trace Amounts of Peroxides In Organic Solvents¹

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1. Scope

1.1 This test method^{2,3} covers organic solvents containing active oxygen in the range from 5 to 80 ppm or higher. By using a special reaction-absorption cell, the test method can be extended to cover the range from 0 to 5 ppm. The test method can be used to determine numerous peroxide classes of varying reactivity such as hydroperoxides, diacyl peroxides, diaroyl peroxides, peresters, and ketone peroxides. The stable di-*tert*-alkyl peroxides do not react under the conditions of analysis.

1.2 Solvents that can be analyzed successfully include saturated and aromatic hydrocarbons, alcohols, ethers, ketones, and esters. In addition, the test method is applicable to olefinic solvents and to certain compounds that contain α, β, and conjugated unsaturation. Solid samples that are soluble in the acetic acid-chloroform solvent also can be analyzed.

1.3 *This standard does not purport to address 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.*

1.4 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid practices, and safety precautions.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water⁴

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵

3. Summary of Test Method

3.1 A sample is dissolved in a mixture of acetic acid and chloroform. The solution is deaerated and potassium iodide reagent solution is added. The mixture is allowed to react in the dark for 1 h, thereby releasing an equivalent amount of iodine.

The absorbance of the solution is measured at 470 nm and the amount of active oxygen present in the sample is determined by reference to a calibration curve prepared from iodine.

3.2 For samples containing 0 to 5 ppm active oxygen, a special reaction-absorption cell is employed. The sample is de-aerated and the reaction is carried out within the cell. Absorbance measurements are made at 410 nm to increase the sensitivity.

4. Significance and Use

4.1 Dilute solutions of peroxides in various organic solvents frequently are used as catalysts or reaction initiators. Peroxides also can be formed through autoxidation in certain classes of compounds including ethers, acetals, dienes, and alkylaromatic hydrocarbons and present a potential safety hazard. This test method provides a procedure for determining the peroxide or active oxygen level.

5. Interferences

5.1 Oxidizing or reducing substances present in the sample will interfere. Colored solutions can be analyzed if an absorbance correction is made.

6. Apparatus

6.1 *Spectrophotometer*—Beckman Model DU or equivalent with matched 1-cm cells.

6.2 *Special Reaction-Absorption Cell* (Fig. 1)—When this cell is used, the regular Beckman cell carriage shall be replaced with the attachment provided for measuring the absorbance in test tubes.

7. Reagents

7.1 *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.

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.01 on General Standards.

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² Banerjee, D. K., and Budke, C. C., *Analytical Chemistry*, ANCHAM, Vol 36, 1964, pp. 792–796.

³ Banerjee, D. K., and Budke, C. C., *Analytical Chemistry*, ANCHAM, Vol 36, 1964, pp. 2367–2368.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

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.