Designation: E 298 – 01

Standard Test Methods for Assay of Organic Peroxides[1]

This standard is issued under the fixed designation E 298; 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 These test methods cover the assay of organic peroxides. Two procedures are given, depending upon the ease of reduction:

1.1.1 *Sodium Iodide, Room Temperature Test Method*, for organic peroxides easy to reduce, and

1.1.2 *Sodium Iodide, 60°C Test Method*, for organic peroxides that are moderately stable.

1.2 *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.*

1.3 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, and safety precautions.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[3]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[3]

3. Significance and Use

3.1 Organic peroxides are widely used as chemical intermediates, catalysts, and initiators. These test methods provide procedures for assaying organic peroxides to determine if they are suitable for their intended use.

4. Purity of Reagents

4.1 *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.

4.2 Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D 1193.

SODIUM IODIDE, ROOM TEMPERATURE TEST METHOD

5. Scope

5.1 This test method covers the assay of organic peroxides that are easily reduced, such as hydroperoxides and diacyl peroxides. Dialkyl peroxides do not react, while peresters and bridge-type peroxides like ascaridole react slowly and incompletely.

5.2 Specific peroxides that can be analyzed by this procedure include the following:

5.2.1 *Diacyl Peroxides*—Benzoyl 2,4-dichlorobenzoyl, *p*-chlorobenzoyl, lauroyl, acetyl, and decanoyl peroxides.

5.2.2 *Dibasic Acid Peroxide*—Succinic acid peroxide.

5.2.3 *Ketone Peroxides*—Cyclohexanone peroxide and methyl ethyl ketone peroxide.

5.2.4 *Alkyl Hydroperoxides*—*tert*-Butylhydroperoxide and 2,5-dimethylhexane-2,5-dihydroperoxide.

6. Summary of Test Method

6.1 A sample is dissolved in a mixture of methylene chloride and acetic acid. A saturated solution of sodium iodide is added and the mixture is allowed to react in the dark at room temperature for 15 min. The liberated iodine is then titrated with standard sodium thiosulfate solution.

7. Interferences

7.1 Conjugated diolefins interfere by absorbing iodine.

8. Apparatus

8.1 *Iodine Flasks*, 250-mL, with stoppers.

NOTE 1—All glassware should be thoroughly cleaned before use.

[1] These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicalsand are the direct responsibility of Subcommittee E15.01 on General Standards.

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

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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