



Designation: E 1228 – 94 (Reapproved 1999)^{ε1}

Standard Test Method for Assay of Peroxy Esters—Catalyzed Iodometric Procedure¹

This standard is issued under the fixed designation E 1228; 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.

^{ε1} NOTE—1.2 was added editorially in May 2000.

1. Scope

1.1 This test method covers the assay of organic peroxides of the peroxy ester type.

NOTE 1—Other test methods for the assay of organic peroxides are given in Test Methods E 298, E 475, and E 755.

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

1.3 The values stated in SI units are to be regarded as the 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. Specific hazards statements are given in Section 9.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis³

E 298 Test Methods for Assay of Organic Peroxides³

E 475 Test Method for Assay of Di-*tert*-Butyl Peroxide by Gas Chromatographic Analysis³

E 755 Test Method for Assay of Dicumyl Peroxide by Liquid Chromatography³

3. Terminology

3.1 Definitions:

3.1.1 *active oxygen*—the oxidizing power present in organic peroxides expressed as oxygen (equivalent weight 8.00).

4. Summary of Test Method

4.1 A sample is dissolved in a mixture of isopropyl alcohol, acetic acid, and cupric chloride. A solution of potassium iodide is added and the mixture is briefly heated, then allowed to react in the dark at room temperature for 30 min. The cupric ion catalyzes the reduction of the peroxide and the liberated iodine is titrated with standard sodium thiosulfate solution.

5. Significance and Use

5.1 Peroxy esters are widely used as chemical intermediates, catalysts, and initiators. This test method provides a procedure for assaying peroxy esters to determine if they are suitable for their intended use.

6. Interferences

6.1 Conjugated diolefins interfere under the conditions of analysis by absorbing iodine.

7. Apparatus

7.1 *Iodine Flasks*, 250-mL capacity, with stoppers.

NOTE 2—All glassware should be cleaned thoroughly with dichromate cleaning solution before use.

7.2 *Beakers*, 1-mL capacity, glass or PTFE.

7.3 *Buret*, 50-mL capacity, graduated in 0.1-mL subdivisions.

7.4 *Water Bath*, maintained at 60°C.

8. Reagents

8.1 *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.

8.2 *Purity of Water*—Unless otherwise indicated, references

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 *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.