



Standard Test Method for Total Peroxides in Acrylonitrile¹

This standard is issued under the fixed designation E 1784; 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 describes a procedure for determining the total peroxide content of acrylonitrile in the range of 0.1 to 0.6 mg H₂O₂/kg acrylonitrile (ppm). Peroxides are an undesirable impurity in acrylonitrile.

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.* Specific hazard statements are given in Section 7.

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 Water²

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals³

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis⁴

3. Summary of Test Method

3.1 Peroxides in the sample react with potassium iodide to form the yellow I₃ ion. The intensity of the color, which is proportional to the concentration of the I₃ ion, is measured at 365 nm. Results are expressed as total peroxides as H₂O₂.

4. Significance and Use

4.1 This test method provides for the determination of total peroxide in acrylonitrile in the range of 0.1 to 0.6 ppm. Only peroxides or other oxidants that react under the conditions of this test method are measured.

5. Apparatus

5.1 *Spectrophotometer*, capable of measuring at 365 nm,

5.2 *Absorption Cells*, 1-cm, borosilicate, matched, and

5.3 *Buret*, 25-mL capacity.

NOTE 1—Photometers and photometric practice described in this test method shall conform to Practice E 60.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ 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 mean Type II or Type III reagent water conforming to Specification D 1193.

6.3 *Potassium Iodide*.

6.4 *Acetic Anhydride*.

6.5 *Hydrogen Peroxide, 30 %*.

6.6 *Acrylonitrile, Peroxide Free*—Prepare by passing acrylonitrile through a 2 cm × 40 cm glass column packed with 50 mL of activated alumina or acid form ion exchange resin.⁶ Flow through the column should be approximately 8 to 10 bed volumes per h. The absorbance obtained by following 9.1 through 9.3 should be less than 0.01. Alternative means of preparation include extraction with 10 % NaOH followed by distillation. For the extraction technique, extract 100 mL of acrylonitrile with 50 mL of 10 % NaOH, follow with triple extraction with water. The volume of peroxide free acrylonitrile required for calibration is 500 mL.

6.7 *Sulfuric Acid Solution, 12 N*—Measure 360 mL of concentrated sulfuric acid (H₂SO₄ sp gr 1.84) into a 500-mL graduated cylinder and slowly add it to 400 mL of water in a 1000-mL beaker while stirring. Rinse the cylinder with water and add the washings to the beaker with water. Mix the

¹ This test method is under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.58 on Acrylonitrile.

Current edition approved March 10, 1997. Published June 1997. Originally published as E 1784 – 96.

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

³ *Annual Book of ASTM Standards*, Vol 03.05.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *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.

⁶ Rohm and Haas Amberlyst 15 has been found to be suitable or an equivalent resin may be used.