



Designation: E 1863 – 97

Standard Test Method for Analysis of Acrylonitrile by Gas Chromatography¹

This standard is issued under the fixed designation E 1863; 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 impurities in, and the purity of, acrylonitrile by flame ionization detector gas chromatography. It is applicable to acrylonitrile in the range of 99 to 100 % purity.

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 Sections 6 and 7.

1.3 Review the current MSDS for detailed information concerning toxicity, first aid procedures, and safety precautions.

2. Referenced Documents

2.1 ASTM Standards:

- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals²
- E 1178 Test Methods for Analysis of Acrylonitrile²

3. Summary of Test Method

3.1 In this test method, a representative sample of acrylonitrile is introduced in a gas chromatograph equipped with a flame ionization detector and a capillary column where specific impurities are separated from the acrylonitrile. The chromatographic area for each impurity is compared to the area of the internal standard, toluene or other suitable known, added to the sample. From response factors of these impurities relative to that of the internal standard and the amount of the standard added, the concentration of the impurities are calculated. Other impurities, such as water, are obtained by use of Test Methods E 1178. The acrylonitrile content is obtained by subtracting the total amount of all impurities from 100.0.

4. Significance and Use

4.1 This test method is designed to obtain acrylonitrile purity on the basis of impurities normally present in acrylonitrile and may be useful for final product inspection and process control.

4.2 This test method will measure the following impurities: acetone, acrolein, acetonitrile, and acetaldehyde. It also will allow calibration of other impurities including benzene, methacrylonitrile, oxazole, propionitrile, cis-crotonitrile, trans-crotonitrile and others where specific impurity standards are available.

5. Apparatus

5.1 *Capillary Gas Chromatograph*—Any gas chromatograph equipped with a flame ionization detector and a split injection system for use with capillary columns that can be operated at the conditions given in Table 1.

5.2 *Column*—The column, such as Supelcowax 10,³ must give satisfactory resolution and proper peak shapes for the components listed in Fig. 1.

5.3 *Recorder/Integrator*—Electronic integration is recommended for this analysis.

5.4 *Syringe*, 10 µL capacity.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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.

6.2 *Carrier Gas*, helium minimum purity 99.995 Mol. %.

6.3 *Hydrogen*, high purity.

6.4 *Air*, hydrogen free, high purity.

6.5 *Toluene*, 99.0 % min purity or other internal standard, previously analyzed free of compounds coeluting with impurities in the sample.

6.6 *Acrylonitrile*, highest purity available.

NOTE 1—High purity acrylonitrile can be obtained by fractional distillation.

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on Product Standards.

Current edition approved March 10, 1997. Published July 1997.

² *Annual Book of ASTM Standards*, Vol 15.05.

³ Supelcowax 10 fused silica capillary column (60 m by 0.32 mm ID by 1 µm film thickness), Supelco Inc., Supelco Park, Bellefonte, PA, has been found suitable.

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.