Designation: E1863 – 18

Standard Test Method for Analysis of Acrylonitrile by Gas Chromatography[1]

This standard is issued under the fixed designation E1863; 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 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Sections 6 and 7.

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

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E1178 Test Methods for Analysis of Acrylonitrile (Withdrawn 2016)[3]

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 E1178. 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-crotononitrile, trans-crotononitrile, 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,[4] 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.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

Current edition approved Oct. 1, 2018. Published October 2018. Originally approved in 1997. Last previous edition approved in 2007 as E1863 – 07, which was withdrawn January 2016 and reinstated in October 2018. DOI: 10.1520/E1863-18.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] A Supelcowax 10 fused silica capillary column (60 m by 0.32 mm ID by 1 µm film thickness), has been found to be suitable, but similar polar wax columns from other vendors may be used after shown to be suitable.

TABLE 1 Typical Instrument Parameters

Detector:	Flame ionization
Column:	
length:	60 m
inside diameter:	0.32 mm
stationary phase:	Supelcowax 10
support:	Fused silica
film thickness, µm:	1.0
Temperature, °C	
injector:	225
detector:	350
oven:	50 initial, hold 25 min, rate 10°/min, 90 final, hold 20 min
Carrier Gas:	Helium
Flow:	20 cm/s
Total chromatograph run time:	54 min
Internal standard:	Toluene
Sample size, µL	2.0
Split ratio	1:100
Retention times, min (refer to Fig. 1 for typical chromatogram)	
Acetaldehyde	6
Acetone	9
Acrolein	10
Benzene	18
Methacrylonitrile	23
Oxazole	29
Propionitrile	29.5
Toluene	30
cis-Crotononitrile	38
trans-Crotononitrile	49

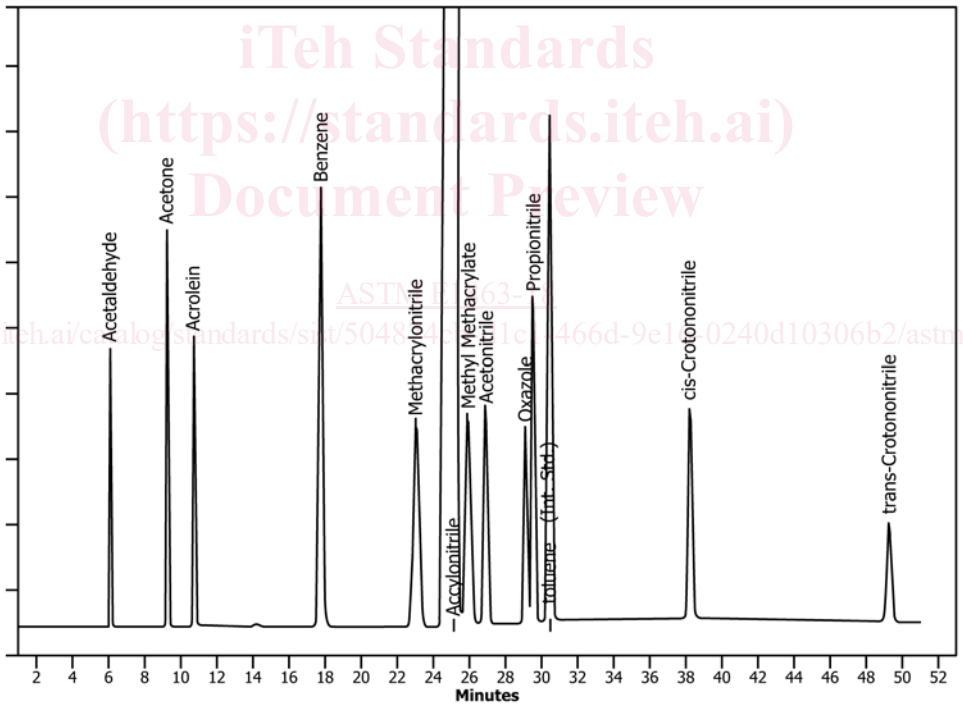

FIG. 1 Typical Chromatogram of Acrylonitrile Calibration Mixture

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