Designation: E1178 – 08

StandardTest Methods for Analysis of Acrylonitrile[1]

This standard is issued under the fixed designation E1178; 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 analysis of acrylonitrile.

1.2 The analytical procedures appear in the following order:

	Sections
Color	7 to 9
Hydrogen Cyanide	10 to 18
p-Methoxyphenol	19 to 28
Water	29 to 32

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

1.5 *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.* See Section 5 for specific hazards statements.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Significance and Use

3.1 These test methods provide for the determination of color and various impurities in acrylonitrile. Color and impurity levels are important factors in many uses of acrylonitrile.

4. Purity of Reagents

4.1 *Purity of Reagents*—Use reagent grade chemicals 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,[4] where such specifications are available. 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D1193.

5. Hazards

5.1 Acrylonitrile is potentially hazardous to human health if not properly handled. Acrylonitrile is a suspected human carcinogen. Use acrylonitrile in a well ventilated hood.

5.2 Acrylonitrile can contribute to a toxic condition in systems of the human body, from inhalation, swallowing, or contact with the eyes or skin. Direct contact with acrylonitrile can cause skin burns as well.

5.3 Acrylonitrile liquid and vapor are absorbed readily into shoe leather and clothing and will penetrate most rubbers, barrier fabrics, or creams. Contact lenses should not be worn in areas where eye contact with acrylonitrile could occur. Use impermeable protective clothing and consult the current MSDS for recommended materials.

[1] This specification is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.16 on Industrial and Specialty Product Standards.

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[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] *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.

***A Summary of Changes section appears at the end of this standard**

6. Photometers and Photometric Practice

6.1 The photometers and photometric practice described in these test methods shall conform to Practice E60.

COLOR

7. Procedure

7.1 Determine the color of the acrylonitrile in accordance with Test Method D1209.

8. Report

8.1 Estimate and report the color of the acrylonitrile to the nearest 1 Pt-Co unit, absolute.

9. Precision and Bias

9.1 The precision and bias of this test method have not been determined.[5]

HYDROGEN CYANIDE

10. Scope

10.1 This test method covers the determination of HCN over the concentration of 0.5 to 10 µg/g in acrylonitrile.

11. Summary of Test Method

11.1 Hydrogen cyanide (HCN) is extracted from the sample as a water soluble salt by means of a solution containing sodium hydroxide, ammonium hydroxide, and potassium iodide. The aqueous extract is then titrated with standard silver nitrate until all the cyanide is complexed as soluble $Ag(CN)2^-$. The first excess of silver, the end point, is indicated visually with the formation of insoluble silver iodide (turbidity). The HCN content is calculated from the quantity of silver nitrate consumed.

12. Significance and Use

12.1 The level of HCN in product acrylonitrile is an indication of product quality and stability. Knowledge of the HCN content can be used in judging quality and monitoring stability.

13. Apparatus

13.1 *Buret,* micro, 10-mL capacity, graduated in 0.02 mL subdivisions.

13.2 *Separatory funnels,* 250-mL capacity.

14. Reagents

14.1 *Potassium Iodide* (KI), crystal.

14.2 *Ammonium Hydroxide* (NH_4OH), concentrated.

14.3 *Sodium Hydroxide* (NaOH), pellets.

14.4 *Caustic Iodide Reagent*—Dissolve 3.6 g of potassium iodide crystals and 44.1 g of sodium hydroxide pellets in approximately 700 mL of water contained in a 1-L volumetric flask. Add 180 mL of concentrated NH_4OH and swirl to mix. Dilute to volume with water, mix thoroughly, and store in a plastic screw-capped bottle. This solution is stable for at least 6 months.

14.5 *Standard Silver Nitrate Solution,* 0.1 meq/mL—Prepare and standardize as described in Practice E200.[6]

14.6 *Standard Silver Nitrate Solution,* 0.01 meq/mL—Pipet 50.0 mL of standard 0.1 *N* $AgNO_3$ solution into a 500-mL volumetric flask. Dilute to volume with water and mix thoroughly.

15. Procedure

15.1 Add 100 mL of caustic iodide to each of three 125-mL Erlenmeyer flasks using a graduated cylinder.

15.2 Slowly titrate each solution with standard 0.01 meq/mL $AgNO_3$ to the same slight persistent opalescence.

15.3 Transfer the contents of two of the flasks into glass-stoppered 250-mL separatory funnels. Retain the third flask for reference. Add 100 mL of sample to each separatory funnel using a graduated cylinder.

15.4 Stopper and shake the separatory funnels for 1 min ±10 s, then allow the layers to separate.

15.5 Drain the lower aqueous layers into the original 125-mL Erlenmeyer flasks.

15.6 Titrate each solution with standard 0.01 meq/mL $AgNO_3$ until an opalescence is obtained that matches the reference solution. Record the volume of 0.01 meq/mL $AgNO_3$ required for each titration and calculate the duplicate values for HCN content.

16. Calculation

16.1 Calculate the HCN content of the sample as follows:

$$\text{HCN, µg/g (m/m)} = \frac{(\text{mL } AgNO_3)(N\ AgNO_3)(0.054) \times 10^6}{(\text{mL sample})(0.806)} \quad (1)$$

where:

0.054 = meq weight of HCN, and
0.806 = density acrylonitrile, g/mL.

$$\text{or HCN, µg/g (m/m)} = \frac{(\text{mL } AgNO_3)(N\ AgNO_3)(66914)}{(\text{mL sample})} \quad (2)$$

17. Report

17.1 Report the µg/g of HCN to two significant figures.

18. Precision and Bias

18.1 The following criteria should be used for judging the acceptability of results (Note 1):

18.1.1 *Repeatability (Single Analyst)*—The coefficient of variation for a single determination has been estimated to be 17.6 % relative at 12 df. The 95 % limit for the difference between two such runs is 49 % relative.

18.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The coefficient of variation of results (each

[5] A round-robin test was conducted with acrylonitrile for color. In each case the value reported was <5 Pt-Co units which is typical for industrial grade acrylonitrile.

[6] This solution may be purchased from Fisher Scientific Co., No. 5572, or an equivalent.