Designation: E 1788 – 96

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Acidity in Acrylonitrile[1]

This standard is issued under the fixed designation E 1788; 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 total acidity as acetic acid in concentrations below 500 ppm 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 8.

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 Water[2]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[3]

3. Summary of Test Method

3.1 The acrylonitrile sample is titrated with alcoholic sodium hydroxide solution to the visual end point.

4. Significance and Use

4.1 This test method is useful for determining low levels of acidity, below 500 ppm in acrylonitrile. The total acidity is calculated as acetic acid.

4.2 Acidity may be present as a result of contamination or decomposition during storage, distribution, or manufacture.

5. Apparatus

5.1 *Buret*, 10 mL, graduated in 0.05 mL subdivision.

6. Purity of Reagents

6.1 Use reagent grade chemicals in all tests. 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.[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 Unless otherwise indicated, references to water mean reagent water conforming to Type II or Type III of Specification D 1193.

7. Reagents

7.1 *Bromthymol Blue Indicator Solution* (1 g/L)—Weigh 100 mg into a 150 mL beaker, add 1.5 mL of 0.1 N NaOH to aid in dissolving, gradually add 100 mL of methanol. Neutralize to a deep green by dropwise addition of 0.02 N NaOH.

7.2 *Sodium Hydroxide Standard Solution* (0.02 N)—Dissolve 0.800 g NaOH in 500 mL of methanol and dilute to 1 L with methanol. Standardize in accordance with the appropriate sections of Test Method E 200.

8. Hazards

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

8.2 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.

8.3 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. Impermeable protective clothing must be used. Consult the current MSDS for recommended materials.

9. Procedure

9.1 To a 250-mL Erlenmeyer flask, add 0.5 mL of bromthymol blue indicator solution.

9.2 Pipet 100 mL of the sample into the 250-mL Erlenmeyer flask. Titrate with the 0.02 N NaOH solution to the first perceptible blue color.

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[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, 1996. Published May 1996.

[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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

10. Calculation

10.1 Calculate the acidity of the sample as follows:

$$\text{Acidity as Acetic Acid, ppm by wt.} = \frac{A \times N \times 60\,000}{B \times D} \quad (1)$$

where:

A = NaOH solution required for titration of sample, mL,
N = normality of the NaOH solution,
B = volume of sample used, mL, and
D = Sp Gr of the sample at the test temperature.

11. Report

11.1 Report the total acidity as acetic acid to the nearest part per million.

12. Precision and Bias

12.1 *Precision*—The following criteria should be used for judging the acceptability of results (see Note 1):

12.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be the absolute value in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is given in Table 1.

TABLE 1 Precision for Total Acidity

Level, ppm	Repeatability			Laboratory Precision			Reproducibility		
	Standard Deviation	df	95 % Limit	Standard Deviation	df	95 % Limit	Standard Deviation	df	95 % Limit
2	0.0000	12	3[A]	0.2887	6	0.8	0.5323	5	1
103	1.3229	10	4	0.7246	5	2	3.5532	4	10

[A] Practical limit, based on reporting results to nearest 1 ppm.

12.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days has been estimated to be the absolute value in Table 1. The 95 % limit for the difference between two such averages is given in Table 1.

12.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be the absolute value in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is given in Table 1.

NOTE 1—The above precision estimates are based on an interlaboratory study of analyses performed in 1991 on two samples of acrylonitrile containing an average total acidity of 2 and 103 ppm, respectively. One analyst in each of seven laboratories performed duplicate determinations and repeated one day later for a total of 56 determinations. Data from one laboratory, however, were excluded from the first sample and two laboratories were excluded from the second sample. Practice E 180 was used in developing these precision estimates.

12.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

13. Keywords

13.1 acidity; acrylonitrile; alcoholic sodium hydroxide; end point; indicator; titrated