



Designation: E224 – 23

Standard Test Methods for Analysis of Hydrochloric Acid¹

This standard is issued under the fixed designation E224; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the analysis of hydrochloric acid.

1.2 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 The analytical procedures appear in the following order:

	Sections
Total Acidity	8 to 16
Baumé Gravity	17 to 26
Sulfated Ash	27 to 34
Iron	35 to 44
Color	45 to 52
Total Sulfur	53 to 59

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this specification. Specific hazards statements are given in Section 5, 30.1, 39.7, and 48.4.*

1.6 *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.*

¹ These test methods are under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and are the direct responsibility of Subcommittee D16.10 on Acids.

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2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

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

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E1 Specification for ASTM Liquid-in-Glass Thermometers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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

E100 Specification for ASTM Hydrometers

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

E300 Practice for Sampling Industrial Chemicals

3. Significance and Use

3.1 These test methods provide for the classification of various grades of hydrochloric acid and for the determination of various impurities. Acid strength and impurity levels are important factors in many uses of hydrochloric acid.

4. Purity of Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used 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,

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.

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

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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type I or Type II reagent water conforming to Specification D1193.

5. Hazards

5.1 Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this test method.

5.2 Hydrochloric acid is a corrosive acid and is dangerous if improperly handled. Avoid any skin contact. Use of safety goggles and gloves highly recommended.

5.3 Clean up all spills immediately by covering the spill with vermiculite or some other inert absorbent material and sweeping into a pan. Dispose of the absorbent by flooding with water and discarding in a suitable container. Flush the area with water.

6. Photometers and Photometric Practice

6.1 Photometers and the photometric practice prescribed in these test methods shall conform to Practice E60.

7. Sampling

7.1 Sample hydrochloric acid in accordance with the appropriate sections of Practice E300 for simple liquids.

7.2 The sample to be analyzed shall be considered to be that sample in a single bottle submitted to the analytical laboratory.

7.3 The size of the sample shall be sufficient to perform all analyses without the reuse of any portion of the sample.

TOTAL ACIDITY

8. Scope

8.1 This test method covers the determination of the total acidity of 27 % to 37 % mass (m/m) hydrochloric acid.

9. Summary of Test Method

9.1 A weighed sample of acid is diluted in water and titrated with standardized 0.5 meq/mL (*N*) sodium hydroxide solution, using phenolphthalein as the indicator.

10. Interferences

10.1 Acids other than hydrochloric and compounds that consume sodium hydroxide will affect the accuracy of this test method.

⁴ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.

11. Apparatus

11.1 *Buret*, 50 mL, Class A.

11.2 *Weighing Bottle*, glass-stoppered, 50 mL.

12. Reagents

12.1 *Phenolphthalein Indicator, Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %), methanol, or isopropanol.⁵

12.2 *Sodium Hydroxide, Standard Solution* (0.5 meq/mL (*N*))—See Practice E200. Correct for differences in temperature in accordance with the following formula:

$$N = N_s + 0.00014 (s - t) \quad (1)$$

where:

N = normality meq/mL (*N*) of NaOH solution at temperature *t*,

N_s = normality meq/mL (*N*) of NaOH solution at temperature *s* during standardization,

s = temperature of NaOH solution during standardization, °C, and

t = temperature of NaOH solution during analysis, °C.

13. Procedure

13.1 Transfer approximately 30 mL of water to a 50 mL glass-stoppered weighing bottle, stopper, and weigh to the nearest 0.1 mg. Rapidly add a convenient size sample, depending upon the acid strength as given in Table 1, stopper immediately, and reweigh. Transfer the sample to a 400 mL beaker containing approximately 50 mL of water and add 3 to 5 drops of phenolphthalein indicator solution. Record the temperature of the 0.5 meq/mL (*N*) NaOH solution, and then titrate the sample to a pink end point. Record the titration to the nearest 0.02 mL.

14. Calculation

14.1 Correct the buret reading for calibration errors, and record as *V* the corrected delivered volume at the recorded temperature.

14.2 Calculate the total acidity as % mass (m/m) of hydrochloric acid as follows:

$$\text{Hydrochloric acid, \% mass (m/m)} = \left(\frac{VN \times 0.03646}{W} \right) \times 100 \quad (2)$$

⁵ This reagent is also described in Practice E200.

TABLE 1 Sample Size For Total Acidity

HCl, % mass (m/m)	Sample Size, g
37	1.9 to 2.3
35	2.0 to 2.4
33	2.2 to 2.6
31	2.3 to 2.8
29	2.5 to 3.0
27	2.7 to 3.2