



Designation: E 224 – 96 (Reapproved 2002)

Standard Test Methods for Analysis of Hydrochloric Acid¹

This standard is issued under the fixed designation E 224; 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 Department of Defense.

1. Scope

1.1 These test methods cover the analysis of hydrochloric acid.

1.2 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

1.3 *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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 5 and 30.1, 39.7, and 48.4.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)³

E 1 Specification for ASTM Thermometers⁴

E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry⁵

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁶

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis⁶

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, where such specifications are available.⁷ 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 D 1193.

5. Hazards

5.1 Hydrochloric acid is a corrosive acid and is dangerous if improperly handled. Avoid any skin contact.

5.2 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 E 60.

7. Sampling

7.1 Sampling of hydrochloric acid is not within the scope of these test methods.

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.

¹ These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and are the direct responsibility of Subcommittee E15.02 on Product Standards.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.

⁶ *Annual Book of ASTM Standards*, Vol 15.05.

⁷ *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.

TOTAL ACIDITY

8. Scope

8.1 This test method covers the determination of the total acidity of 27 to 37 % hydrochloric acid.

9. Summary of Test Method

9.1 A weighed sample of acid is diluted in water and titrated with standardized 0.5 *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.

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.⁸

12.2 *Sodium Hydroxide, Standard Solution* (0.5 *N*)—See Practice E 200. Correct for differences in temperature in accordance with the following formula:

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

where:

N = normality of NaOH solution at temperature *t*,

N_s = normality of NaOH solution at temperature *s* during standardization,

s = temperature of NaOH solution during standardization, 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

TABLE 1 Sample Size For Total Acidity

HCl, %	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

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 *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 percentage of hydrochloric acid as follows:

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

where:

V = corrected mL of NaOH solution required for titration of the sample,

N = normality of the NaOH solution, and

W = sample used, g.

15. Report

15.1 Report the percentage of hydrochloric acid to the nearest 0.01 %.

16. Precision and Bias

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

16.1.1 *Repeatability (Single Analyst)*—The coefficient of variation for a single determination has been estimated to be 0.133 % relative at 50 df. The 95 % limit for the difference between two such runs is 0.37 % relative.

16.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), Formerly Called Repeatability*—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.170 % relative at 25 df. The 95 % limit for the difference between two such averages is 0.48 % relative.

16.1.3 *Reproducibility (Multilaboratory)*—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.285 % relative at 7 df. The 95 % limit for the difference between two such averages is 0.80 % relative.

NOTE 1—These precision estimates are based on an interlaboratory study of analyses performed in 1963 on three samples containing approximately 28, 31, and 38 % hydrochloric acid. One analyst in each of ten laboratories performed duplicate determinations and repeated one day later, for a total of 120 determinations.⁹ Practice E 180 was used in developing these precision estimates.

16.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

BAUMÉ GRAVITY

17. Scope

17.1 This test method covers the determination of the Baumé gravity of hydrochloric acid by means of a glass

⁸ This reagent is also described in Practice E 200.

⁹ Details of the interlaboratory study are available from ASTM International Headquarters. Request RR: E15 - 1046.

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hydrometer in the range from 17.5 to 23° Baumé. The Baumé gravity is determined at 15.5°C (60°F).

18. Terminology

18.1 Definition:

18.1.1 *Baumé gravity*—a unit of density based on specific gravity and defined by the following equation:

$$\text{Baumé gravity} = 145 - [145/(\text{sp gr } 15.5/15.5^{\circ}\text{C } (60/60^{\circ}\text{F}))] \quad (3)$$

19. Summary of Test Method

19.1 A sample of hydrochloric acid is placed in a hydrometer cylinder and when the temperature is constant, the Baumé gravity is read from the glass hydrometer.

20. Significance and Use

20.1 The Baumé gravity is used to classify various grades of hydrochloric acid.

21. Apparatus

21.1 *Hydrometer*,¹⁰ streamline or torpedo design, precision grade, for liquids heavier than water in ranges from 17.5 to 23°Bé. The total length shall be approximately 12 in. (305 mm) divided to 0.1°Bé over a 6-in. (152-mm) (approximate) scale and standardized at 15.5/15.5°C (60/60°F) with a tolerance of 0.1°Bé throughout. The modulus is as follows:

$$\text{Bé} = 145 - [145/\text{sp gr } 15.5/15.5^{\circ}\text{C } (60/60^{\circ}\text{F})] \quad (4)$$

Each of the hydrometers shall show on the scale the modulus (or formula).

21.2 *Thermometer*, having a range from – 2 to + 80°C (30 to 180°F) and conforming to the requirements for Thermometer 15C (15F) in accordance with Specification E 1.

21.3 *Cylinder, Hydrometer*, glass with or without lip, diameter 38 to 40 mm, height 325 to 375 mm.

22. Temperature of Test

22.1 Baumé gravity shall be determined at 15.5 ± 0.3°C (60 ± 0.5°F).

23. Procedure

23.1 Rinse a clean hydrometer cylinder with the sample to be tested, add the sample, and adjust the temperature to 15.5 ± 0.3°C (60 ± 0.5°F). Place the cylinder in a vertical position in a location free of air currents. Insert the hydrometer when it has come to rest, floating freely, and the temperature is 15.5°C (60°F). The correct reading is that point of the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

24. Calculation

24.1 Calculate the specific gravity for use in the determination of iron using the following equation:

$$\text{sp gr} = \frac{145}{(145 - \text{Bé gravity})} \quad (5)$$

25. Report

25.1 Report the Baumé gravity to the nearest 0.1 unit.

26. Precision and Bias

26.1 The following criteria should be used for judging the acceptability of results (see Note 2):

26.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.048 unit absolute at 48 df. The 95 % limit for the difference between two such runs is 0.1 unit absolute.

26.1.2 *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 0.046 unit absolute at 24 df. The 95 % limit for the difference between two such averages is 0.1 unit absolute.

26.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.084 unit absolute at 7 df. The 95 % limit for the difference between two such averages is 0.2 unit absolute.

NOTE 2—These precision estimates are based on an interlaboratory study of analyses performed in 1963 on three samples having Baumé gravities of approximately 18, 20, and 23 units. One analyst in each of nine laboratories performed duplicate determinations and repeated one day later, for a total of 108 determinations.⁹ Practice E 180 was used in developing these precision estimates.

26.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

SULFATED ASH

27. Scope

27.1 This test method covers the gravimetric determination of material not volatile after treatment with sulfuric acid. The lower limit of determination of sulfated ash is 0.001 %.

28. Summary of Test Method

28.1 A weighed sample of acid, to which sulfuric acid has been added, is evaporated, ignited, and the residue weighed.

29. Apparatus

29.1 *Evaporating Dish*, platinum or high-silica glass, 150-mL.

29.2 *Muffle Furnace*, maintained at 800 ± 25°C (1472 ± 45°F).

29.3 *Crucible Tongs*.

30. Reagent

30.1 *Sulfuric Acid (1 + 1)*—Add slowly with stirring 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) to 1 volume of water. (**Warning**—Use goggles when preparing this solution.)

¹⁰ Suitable hydrometers are available from Walter H. Kessler, Inc., 160 Hicks St., Westbury, L.I., NY 11590.