



Standard Test Methods for Analysis of Sulfuric Acid¹

This standard is issued under the fixed designation E 223; 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 sulfuric acid.
- 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 The analytical procedures appear in the following order:

	Sections
Total Acidity	8 to 16
Baumé Gravity	17 to 26
Nonvolatile Matter	27 to 33
Iron	34 to 43
Sulfur Dioxide	44 to 51
Arsenic	52 to 61

1.4 *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.*

2. Referenced Documents

- 2.1 *ASTM Standards:*²
 - D 1193 Specification for Reagent Water
 - E 1 Specification for ASTM Liquid-in-Glass Thermometers
 - E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry
 - E 100 Specification for ASTM Hydrometers
 - E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals
 - E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

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 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. Significance and Use

3.1 These test methods provide for the classification of various grades of sulfuric acid and for the determination of various impurities. Acid strength and impurity levels are important factors in many uses of sulfuric 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.³ 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 Sulfuric acid is a strong corrosive acid and is dangerous if improperly handled. Avoid any skin or eye 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 used in these test methods shall conform to Practice E 60.

7. Sampling

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

³ *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.

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 75 to 99 % mass (m/m) sulfuric acid. Two test methods are given for weighing the sample, namely, the Dely tube and the snake tube test methods.

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 sulfuric and compounds that consume sodium hydroxide will affect the accuracy of this test method.

11. Apparatus

11.1 *Dely Tube (Fig. 1) or Snake Tube (Fig. 2).*⁴

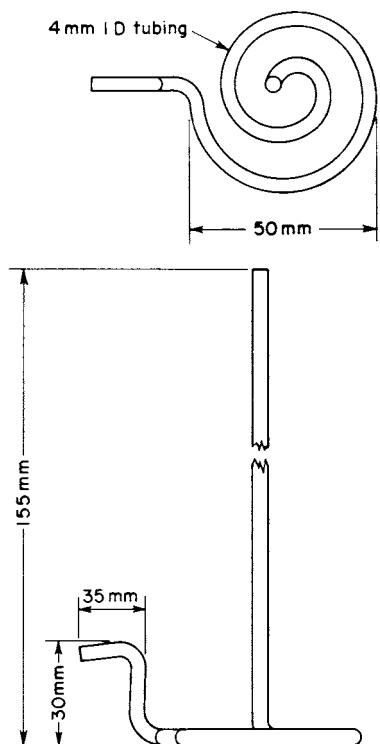


FIG. 1 Dely Tube

11.2 *Buret, 100-mL, Class A, bulb-type.*

⁴ The sole source of supply of the Suitable Dely and snake tubes known to the committee at this time is Corning Glass Works, Corning, NY. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

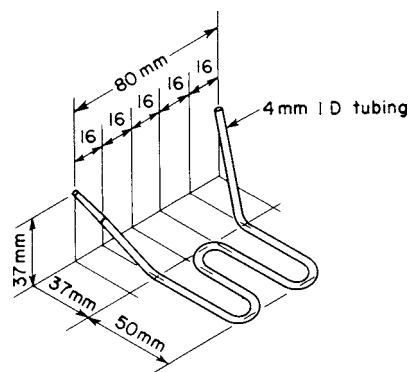


FIG. 2 Snake Tube

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 E 200.

13. Procedure

13.1 *Dely Tube Test Method*—Invert the sample bottle several times. (Hold the stopper in tight.) Insert the long arm of a dry, weighed Dely tube and withdraw by suction a convenient size sample depending upon the acid strength as given in Table 1 (Note 1). Invert the Dely tube and wipe the acid from the long arm with disposable tissue several layers thick. Discard the tissue immediately to avoid burning the fingers. Reweigh to the nearest 0.0001 g and record the weight of the sample. Incline the tube so that the acid runs back nearly to the bend of the short arm. Attach the short arm to an elevated water reservoir by means of a rubber tube closed near the lower end with a pinch clamp. Insert the long arm of the Dely tube into 400-mL glass beaker containing approximately 100 mL of water. Open the pinch clamp and flush the sample into the beaker. Continue the flow of water until all acid is washed from the Dely Tube (Note 2 and Note 3). Wash the long end of the Dely tube, collecting the washings in the beaker. 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.

NOTE 1—The Dely tube can be marked at points equivalent to weights given in Table 1.

⁵ This reagent is also described in Practice E 200.

TABLE 1 Sample Size for Total Acidity

H ₂ SO ₄ , % mass (m/m)	Sample Size, g
98	1.9 to 2.2
94	2.0 to 2.3
90	2.1 to 2.4
85	2.2 to 2.6
80	2.3 to 2.7
77	2.4 to 2.8
75	2.5 to 2.9

NOTE 2—The presence of acid in the Dely tube may be detected by coloring the water in the reservoir with phenolphthalein indicator and the minimum amount of dilute NaOH solution that will produce a slight pink. The water flowing through the tube is discolored as long as acid is present, and the appearance of a pink color indicates the absence of acid.

NOTE 3—The acid and water are separated by a bubble of air.

13.2 *Snake Tube Test Method*—Invert the sample bottle several times. (Hold the stopper in tight). Insert the capillary end of a dry, weighed snake tube and withdraw by suction a convenient size sample depending upon the acid strength as given in Table 1. Invert the tube so that the double bend is in a horizontal position. Wipe the acid from the capillary with disposable tissue several layers thick. Discard the tissue immediately to avoid burning the fingers. Reweigh to the nearest 0.0001 g and record the weight of the sample. Submerge the capillary of the tube in approximately 100 mL of water contained in the 400-mL beaker. Force the weighed sample from the tube by a stream of water from a wash bottle by placing the delivery tip in the exposed end of the snake tube (Note 4). Wash the tube with 50 to 70 mL of water. Remove the tube and wash the outside free of acid. Swirl the contents of the beaker gently while washing. Accumulate all washings in the beaker 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.

NOTE 4—Do not introduce the water into the snake tube too rapidly, as this will cause spattering.

14. Calculation

14.1 If necessary, correct the buret reading for calibration errors and record the volume of titrant as V and the temperature as t .

14.2 Correct the normality of the sodium hydroxide standard solution for any difference in temperature between time of standardization and time of use according to the following equation:

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

where:

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

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

s = temperature of NaOH solution during standardization, and

t = temperature of NaOH solution during analysis.

14.3 Calculate the total acidity as % mass (m/m) of sulfuric acid as follows:

$$\text{Sulfuric acid, \% mass (m/m)} = \frac{(VN \times 0.04904)}{W} \times 100 \quad (2)$$

where:

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

N = meq/mL(N) normality of the NaOH solution, and

W = grams of sample used.

15. Report

15.1 Report the % mass (m/m) of sulfuric acid to the nearest 0.01 % mass(m/m).

16. Precision and Bias

16.1 The following criteria should be used for judging the acceptability of results (see Note 5):

16.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.069 % mass (m/m) absolute at 56 df. The 95 % limit for the difference between two such runs is 0.19 % mass (m/m) absolute.

16.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.104 % mass (m/m) absolute at 28 df. The 95 % limit for the difference between two such averages is 0.29 % mass (m/m) absolute.

16.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.124 % mass (m/m) absolute at 7 df. The 95 % limit for the difference between two such averages is 0.35 % mass (m/m) absolute.

NOTE 5—These precision estimates are based on an interlaboratory study of analyses performed in 1963 on three samples containing approximately 80, 90, and 95 % mass (m/m) sulfuric acid. One analyst in each of ten laboratories performed duplicate determinations and repeated one day later, for a total of 120 determinations.⁶ Practice E 180 was used in developing these precision estimates.

16.2 Since there is no accepted reference material for determining the bias for measuring the total acidity of sulfuric acid, the bias of this test method has not been determined.

BAUMÉ GRAVITY

17. Scope

17.1 This test method covers the determination of the Baumé gravity of concentrated sulfuric acid by means of a glass hydrometer in the range from 57 to 66.2° Baumé. The Baumé gravity is determined at 15.5°C (60°F). This test method is not applicable to readings above 66.2 Baumé gravity units.

18. Definition

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

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

19. Summary of Test Method

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

⁶ Details of the interlaboratory study are available from ASTM International Headquarters. Request RR: E15-1047.