



Designation: **E 1584 – 9500**

## Standard Test Method for Assay of Nitric Acid<sup>1</sup>

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### 1. Scope

1.1 This test method covers determination of the assay of nitric acid by total acidity. This test method is suitable for concentrations between approximately 50 and 70 %, calculated as nitric acid.

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

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

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 precautionary statements are given in Section 8.

### 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water<sup>2</sup>

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<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.01 on Acids-Product Standards.

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E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>3</sup>

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis<sup>3</sup>

E 300 Practice for Sampling Industrial Chemicals<sup>3</sup>

### 3. Summary of Test Method

3.1 A weighed sample of acid is diluted in water and titrated with 1.0 *N* sodium hydroxide solution, using phenolphthalein as the end-point indicator.

### 4. Significance and Use

4.1 This test method provides a means for assaying nitric acid, based on total acidity. The concentration of nitric acid is important in many of the uses of nitric acid, including specification compliance and manufacturing control.

### 5. Interferences

5.1 Acids other than nitric, and compounds that consume sodium hydroxide, will yield erroneously high results.

### 6. Apparatus

6.1 *Erlenmeyer Flask*, 250 mL, glass stoppered.

6.2 *Buret*, 50 mL, Class A.

NOTE 1—A digital buret capable of measuring volumes to the nearest 0.01 mL may be used in place of a conventional buret.

### 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or Type III of Specification D 1193.

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

7.4 *Sodium Hydroxide, Standard Solution* (1.0 *N*)— Prepare and standardize in accordance with Practice E 200, remembering to record the temperature of the NaOH solution during the standardization to the nearest °C.

### 8. Safety Precautions

8.1 Nitric acid is dangerous if handled improperly. It is a strong corrosive acid and a strong oxidizer. Contact with other material may cause fire. Avoid inhalation of fumes or mist and any contact with skin or eyes.

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

### 9. Sampling

9.1 Sampling of nitric acid is not within the scope of this test method. General guidelines for sampling may be found in Practice E 300.

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

### 10. Procedure

10.1 Tare to the nearest 0.1 mg a glass stoppered 250-mL Erlenmeyer flask containing approximately 15 mL of water.

10.2 Remove the stopper. Using a suitable pipet, quickly add an appropriate volume of sample (depending on the acid strength as given in Table 1), while keeping the tip of the pipet just above the surface of the liquid. Immediately replace the stopper, and reweigh the flask to the nearest 0.1 mg to obtain the weight of the sample.

NOTE 2—A disposable dropping pipet is convenient for the sample transfer.

10.3 Remove the stopper, add 50 mL of water and four drops of phenolphthalein indicator solution to the flask, and swirl to mix the contents. Record the temperature of the 1.0 *N* NaOH solution, and then titrate the sample to a pale pink end point. Record the volume of the titrant to the nearest 0.01 mL.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>4</sup> *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.

<sup>5</sup> This reagent is also described in Practice E 200.

**TABLE 1 Sample Size for Assay of Nitric Acid<sup>A</sup>**

HNO <sub>3</sub> , %	Sample Size, mL	Nominal Weight, g
70	2.5 ± 0.2	3.6
65	2.8 ± 0.2	3.9
60	3.1 ± 0.2	4.3
55	3.4 ± 0.2	4.6
50	3.8 ± 0.2	5.0

<sup>A</sup>In some cases, where the concentration range of the sample is unknown, a preliminary test may be required to determine the proper size test sample to use.

## 11. Calculation

11.1 If necessary, correct the buret reading for calibration errors, and record the volume of the titrant as  $V$  and its temperature during the titration as  $t$ .

11.2 Correct the normality of the sodium hydroxide standard solution for any difference in temperature between time of standardization and time of use in accordance with the following equation:

$$N = N_s + 0.00035 (s - t) \quad (1)$$

where:

$N$  = normality of NaOH solution at time of titration,

$N_s$  = normality of NaOH solution at time of standardization,

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

$t$  = temperature of NaOH during titration °C.

11.3 Calculate the assay as percent nitric acid as follows:

$$\text{nitric acid, \% by weight} = \frac{VN \times 6301}{W} \quad (2)$$

where:

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

$N$  = normality of the NaOH solution corrected for temperature at time of titration, and

$W$  = sample used, g.

## 12. Report

12.1 Report the percent of nitric acid to the nearest 0.01 %.

## 13. Precision and Bias

13.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 3):

13.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0914 % absolute at 54 df. The 95 % limit for the difference between two such runs is 0.26 % absolute.

13.1.2 *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 0.0892 % absolute at 27 df. The 95 % limit for the difference between two such averages is 0.25 % absolute.

13.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.1720 % absolute at 8 df. The 95 % limit for the difference between two such averages is 0.48 % absolute.

NOTE 3—The preceding precision estimates are based on an interlaboratory study of analyses performed in 1993 to 1994 on three samples of nitric acid with average assays of 56.49 %, 65.54 %, and 69.65 %. One analyst in each of nine laboratories performed duplicate determinations and repeated one day later, for a total of 108 determinations.<sup>6</sup> Practice E 180 was used in developing these precision estimates.

13.2 *Bias*—The bias of this test method cannot be determined unless a suitable reference material becomes available.

## 14. Keywords

14.1 assay; nitric acid; titrimetric method

<sup>6</sup> Supporting data are available from ASTM Headquarters. Request RR: E15-1041.

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