



Designation: E 358 – 95

Standard Test Methods for Analysis of Phosphoric Acid¹

This standard is issued under the fixed designation E 358; 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 furnace grade phosphoric acid.

1.2 The analytical procedures appear in the following order:

Assay by Specific Gravity	Sections
Iron	8-16
	17-26

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

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:*

D 1193 Specification for Reagent Water²

E 1 Specification for ASTM Thermometers³

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals⁴

E 100 Specification for ASTM Hydrometers³

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis⁵

3. Significance and Use

3.1 These test methods provide for the classification of various grades of phosphoric acid and for the determination of

trace amounts of iron. The acid strength and iron level are important factors in many uses of phosphoric acid.

4. 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.⁶ 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 *Safety Precautions:*

5.1.1 Phosphoric acid is a corrosive acid and is dangerous if improperly handled. Avoid any skin contact.

5.1.2 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 photometric practice prescribed in these test methods shall conform to Practice E 60.

7. Sampling

7.1 Sampling of phosphoric 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 14.03.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

⁶ *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.

ASSAY BY SPECIFIC GRAVITY

8. Scope

8.1 This test method covers the determination of H_3PO_4 in furnace-grade phosphoric acid only, by sp gr at 25°C/15.5°C.

9. Summary of Test Method

9.1 The sample of phosphoric acid is placed in a hydrometer cylinder, and the specific gravity is read from a glass hydrometer placed in the jar. The temperature of the acid is noted and proper corrections applied to the reading.

10. Significance and Use

10.1 The specific gravity can be used to determine the strength of phosphoric acid made from furnace-grade phosphorus.

11. Apparatus

11.1 *Hydrometer*, streamline or torpedo design, precision grade for liquids heavier than water in ranges suitable to the acid strength tested. For specifications, refer to Specification E 100.

ASTM Hydrometer No.	Range	For
136H-62	1.550 to 1.600	75 % acid
137H-62	1.600 to 1.650	80 % acid
138H-62	1.650 to 1.700	85 % acid

Each unit is equivalent to 0.001 sp gr. The distance between each unit is approximately 1.5 mm; overall length is approximately 280 mm (11 in.). The correction factor for each hydrometer should be determined by testing in acid of known specific gravity at 25°C/15.5°C as determined in Section 12 and applied to the hydrometer readings.

11.2 *Thermometer*—ASTM Gravity Thermometer, having a range from -20 to +102°C, and conforming to the requirements of Thermometer 12C as prescribed in Specification E 1.

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

11.4 *Pycnometer*,⁷ either 25 or 50-mL capacity of a type either with or without a built-in thermometer. The latter type is superior for precise work.

12. Calibration of Hydrometer

12.1 Procedure:

12.1.1 Thoroughly clean the pycnometer, rinse with a volatile solvent such as acetone or methanol, and sparge with dry air.

12.1.2 Weigh the clean, dry, assembled pycnometer on an analytical balance and record the weight.

12.1.3 Fill the pycnometer to just above the ground-glass joint with freshly boiled reagent water that has been cooled to a temperature below 25°C.

12.1.4 Carefully insert the top of the pycnometer so that no air remains entrapped. If using a vertical capillary pycnometer,

the water will rise in the capillary and overflow. If using a sidearm capillary, the excess water will overflow from the small side arm capillary.

12.1.5 Place the pycnometer in a water bath set at 25°C. In the case of the vertical capillary pycnometer, allow to remain ½ h. If using a sidearm capillary pycnometer, allow to remain until the temperature in the pycnometer is 25°C.

12.1.6 Remove the pycnometer from the water bath, cool to below 25°C, and wipe off the excess water using a chamois or a soft paper towel and lens tissue. Be careful, when wiping, not to wipe directly over the capillary opening because of the capillary action of the paper toweling.

12.1.7 Carefully place the pycnometer on the balance pan and weigh. Avoid getting any moisture from the fingers on the glass. Record the weight.

12.1.8 Calculate the volume of the pycnometer at 25°C.

12.1.9 Repeat 12.1.1-12.1.8. The calculated volumes should agree within ±0.002 mL.

12.1.10 Using phosphoric acid of proper strength instead of reagent water, repeat 12.1.1-12.1.7 twice. The weights of phosphoric acid obtained should agree within ±0.002 g.

12.1.11 Determine two specific gravity readings on the acid using the hydrometer to be standardized. These values should agree within ±0.0002 units. Correct for temperature to 25°C/15.5°C.

12.1.12 If the entire range of the hydrometer is to be used, the hydrometer calibration should be made at two or more points.

12.2 Calculation:

12.2.1 Calculate the volume of the pycnometer as follows:

$$\text{Volume of pycnometer} = (W_w - W_o)/D \quad (1)$$

where:

W_w = weight of pycnometer plus water, g,

W_o = weight of empty pycnometer, g, and

D = density of water at 25°C = 0.99707, g/mL.

12.2.2 Calculate the density of acid at 25°C as follows:

$$\text{Density of acid at 25°C} = (W_a - W_o)/V \quad (2)$$

where:

W_a = weight of pycnometer plus acid, g,

W_o = weight of empty pycnometer, g, and

V = volume of pycnometer at 25°C, mL.

12.2.3 Calculate the specific gravity of acid, 25°C/15.5°C, as follows:

$$\text{Sp gr of acid, 25°C/15.5°C} = A/W \quad (3)$$

where:

A = density of acid at 25°C, and

W = density of water at 15.5°C.

12.2.4 Hydrometer correction is the number of specific gravity units that must be added to or subtracted from the hydrometer reading to make it equal to the specific gravity obtained by the pycnometer.

12.2.5 Always handle the pycnometer carefully. Any moisture from the fingers will cause an incorrect weight. As an aid in handling, fold a small piece of paper toweling into a narrow strip, place around the neck of the pycnometer, and handle by the extended paper tabs.

⁷ Either a vertical capillary pycnometer (such as Sargent-Welch No. S-9205) or a sidearm capillary pycnometer (such as Sargent-Welch No. S-9195) has been found satisfactory for this purpose.