



Designation: E 357 – 98

Standard Test Method for Phosphorus in Organic Compounds by Sodium Peroxide Bomb Ignition¹

This standard is issued under the fixed designation E 357; 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 This test method² covers the determination of phosphorus in organic compounds by sodium peroxide bomb ignition. It is applicable to samples of organic materials containing more than 1.0 % phosphorus.

1.2 The values stated in SI units are to be regarded as the standard.

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 precautionary statements are given in Section 8, and Note 1, Note 2, and Note 6.

1.4 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, and safety precautions.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water³

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

3. Summary of Test Method

3.1 A sample is heated in a closed bomb with sodium peroxide in the presence of an accelerator mixture consisting of potassium nitrate and sucrose. The phosphorus is converted to phosphate. The melt is dissolved in water and acidified with nitric acid. Phosphorus is determined gravimetrically on an

aliquot of the solution by precipitation as quinolinium phosphomolybdate, $(C_9H_7N)_3H_3PO_4 \cdot 12MoO_3$.

4. Significance and Use

4.1 This test method is applicable to the determination of phosphorus in organic materials containing at least 1.0 % phosphorus. It can be used as an assay procedure for known phosphorus organic compounds.

5. Interferences

5.1 Interfering substances include elements such as arsenic which form insoluble precipitates with the quinoline molybdate reagent, and those which yield insoluble phosphates. Interference from silica is reduced by addition of citric acid to the reagent. Citric acid forms a complex with molybdic acid, of such stability that its reaction with silicic acid is avoided.

6. Apparatus

6.1 *Sodium Peroxide Bomb Apparatus*, consisting of a 22-mL fusion cup of 99 % nickel, a 99 % nickel cup cover, lead gaskets, a bomb body and screw cap, a wrench, a bomb socket, and a measuring dipper holding approximately 15 g of sodium peroxide. A less expensive nickel plated brass cup cover may be substituted.⁵

6.2 *Safety Shield*—Any heavy-duty commercially available shield should suffice. The safety shield used must confine the results of an explosion in the event of bomb body rupture during flame ignition.

6.3 *Burner*, blast, for use with natural gas and oxygen.

6.4 *Capsules*, gelatin, Size No. 0.

6.5 *Crucibles*, Gooch, Coors porcelain, with perforated bottoms, Size No. 3, 25-mL capacity.

6.6 *Filter Pads*, glass-fiber, 2.1-cm diameter.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

¹ 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 General Standards.

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² Official Methods of Analysis of the Association of Official Agricultural Chemists, Tenth Ed., 1965, p. 12, and Perrin, C. H., *Journal of Official Agricultural Chemists*, JOACA, Vol 41, 1959, p. 758.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ Available from Parr Instrument Co., 211 Fifty-Third St., Moline, IL., Catalog No. 2102. The 22-mL fusion cup is part No. N-200.