



Standard Test Methods for Phosphorus in Lubricating Oils and Additives¹

This standard is issued under the fixed designation D 1091; 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 test method has been adopted for use by government agencies to replace Method 5661 of Federal Test Method Standard No. 791b

1. Scope*

1.1 These test methods cover the determination of phosphorus in unused lubricating oils and lubricating oil additives and their concentrates. The test methods are not restricted with respect to the type of phosphorus compounds that may be present—for example, trivalent or pentavalent phosphorus compounds, phosphines, phosphates, phosphonates, phosphorus sulfides, etc.—since all are quantitatively converted to an aqueous solution of orthophosphate ion by oxidation of the sample during the course of analysis.

1.2 *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.*

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

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 3231 Test Method for Phosphorus in Gasoline³
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³
- D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³
- D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁴

3. Summary of Test Method

3.1 Organic material in the sample is removed and the phosphorus is converted to phosphate ion by oxidation with

sulfuric acid, nitric acid, and hydrogen peroxide. One of two procedures is then followed:

	Sections
Photometric (Molybdivanado) Method	7-18
Gravimetric Method	7-11 and 19-25

3.2 The photometric method is used where the phosphorus content is estimated to be under 2 %, and the gravimetric method is used for phosphorus contents of 2 % or over.

4. Significance and Use

4.1 Knowledge of the phosphorus content, and thus the phosphorus-containing additives, in a lubricating oil or additive can be used to predict performance characteristics. This test method is suitable for most applications requiring the determination of phosphorus.

5. Purity of Reagents

5.1 *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.⁵ 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.

5.2 *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.

6. Sampling

6.1 Obtain samples in accordance with the instructions in Practices D 4057 or D 4177.

¹ These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the responsibility of Subcommittee D02.03 on Elemental Analysis.

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

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ *Annual Book of ASTM Standards*, Vol 05.03.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D.C. 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.