



Standard Test Method for Total Nitrogen In Lubricating Oils and Fuel Oils By Modified Kjeldahl Method¹

This standard is issued under the fixed designation D 3228; 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 method is applicable for the determination of nitrogen in lubricating oils when present in the concentration from 0.03 to 0.10 mass %, and for the determination of nitrogen in fuel oils when present in the concentration from 0.015 to 2.0 mass %. This test method is also applicable to the analysis of additive concentrates and additive packages.

NOTE 1—This test method may not be applicable to certain materials containing N–O or N–N linkage. However, the samples used in the cooperative program to establish the precision of the test method were compounded with currently available ashless additives containing nitrogen. Complete recovery of the nitrogen present in these additives was obtained.

1.2 The values given in acceptable 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. For specific precautionary statements, see Note 2, Note 3, Note 5, and Note 6.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis⁴

3. Summary of Test Method

3.1 The sample is digested in a mixture of concentrated sulfuric acid, potassium sulfate, mercuric oxide, and copper

sulfate. After digestion, sodium sulfide is added to precipitate the mercury, and the mixture is made alkaline with caustic. Nitrogen, now in the form of ammonia, is distilled into a boric acid solution. The ammonia is titrated with standard sulfuric acid using methyl purple as an indicator.

4. Significance and Use

4.1 The concentration of nitrogen is a measure of the presence of nitrogen-containing additives. Knowledge of its concentration can be used to predict performance.

5. Apparatus

5.1 *Buret*, 50-mL, graduated in 0.1-mL subdivisions, one for each titrant. Other size burettes may also be used.

5.2 *Flask*, Erlenmeyer, 300-mL. Other sizes are also acceptable.

5.3 *Heater*, electrical or gas.

5.4 *Kjeldahl Distillation Apparatus*.

NOTE 2—Commercially available semi-automatic Kjeldahl apparatus are acceptable. In such cases manufacturer prescribed sizes of burettes and flasks may be used.

5.5 *Kjeldahl Flask*, at least 500-mL volume.

6. Reagents

6.1 *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.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.

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

6.3 *Boric Acid Solution* (40 g/L)—Dissolve 40 g of boric

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct 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 15.05.

⁵ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD. Other commercial catalyst reagent mixtures that provide equivalent results, such as copper and titanium compounds, are also acceptable.