



Standard Test Method for Alkyl Nitrate in Diesel Fuels by Spectrophotometry¹

This standard is issued under the fixed designation D 4046; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Warning notes were placed in the text in April 2000.

1. Scope

1.1 This test method describes a procedure for the determination of 0.03 to 0.30 volume % of alkyl nitrate in diesel fuels.

1.2 This test method can be used for the determination of any alkyl nitrate in diesel fuel provided that standards used for calibration contain the same alkyl nitrate ester as the test specimens to be analyzed.

1.3 The preferred units are acceptable SI units.

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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The determination is based on the simultaneous hydrolysis of the ester in 65 % sulfuric acid solution and nitration of *m*-xylenol by the nitric acid liberated. The nitroxylenol is extracted from the reaction mixture and reacted with sodium hydroxide to form the yellow salt. The color is measured spectrophotometrically at 452 nm, and the concentration of alkyl nitrate is determined by reference to the slope of the calibration curve.

4. Significance and Use

4.1 This test method can be used to determine the amount of alkyl nitrate that has been added to diesel fuel to improve Cetane Number. The test method is applicable as a basis for judging compliance with specifications covering any alkyl nitrate.

5. Interferences

5.1 Other nitrate esters, inorganic nitrate ions, and nitrogen oxides will interfere.

6. Apparatus

6.1 *Absorption Cells*, matched, having a 1.000 ± 0.002 -cm path length.

6.2 *Separatory Funnel*, Squibb-type glass stoppered with TFE-fluorocarbon stopcock, 125-mL capacity.

6.3 *Shaking Machine*, automatic, capable of multiple samples and 250 oscillations/min.

6.4 *Spectrophotometer*, capable of measuring absorption in the region of 452 ± 5 nm.

6.5 *Volumetric Flask*, 10- and 100-mL capacity.

6.6 *Measuring Pipet*, 1-mL, 3-mL, 4-mL, and 10-mL.

6.7 *Buret*, 10-mL capacity.

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.³ 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 conforming to Type II of Specification D 1193.

7.3 *Acetic Acid*, reagent grade.

7.4 *Alkyl Nitrate*.

7.5 *Alkyl Nitrate, Standard Solution*—Pipet 3.0 mL of the alkyl nitrate to be determined into a 100-mL volumetric flask and dilute to volume with diesel fuel.

¹ This test method is under the jurisdiction of ASTM Committee D02 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 *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.