



Designation: ~~D1839~~—~~11~~ D1839 – 14

Standard Test Method for Amyl Nitrate in Diesel Fuels¹

This standard is issued under the fixed designation D1839; 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.

1. Scope*

1.1 This test method covers the determination of 0.1 to 0.5 volume % amyl nitrate in diesel fuels.

NOTE 1—This test method has been used for the determination of hexyl nitrate in diesel fuels, but has not been cooperatively tested for such samples. For the determination of hexyl nitrate, use standards containing nitrate esters of primary hexanol. Use a density of 0.97 in calculating results.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

3. Summary of Test Method

3.1 This test method is based on the simultaneous hydrolysis of the ester in 62.5 % sulfuric acid and nitration of *m*-xyleneol by the nitric acid liberated. The nitroxyleneol is distilled from the reaction mixture and reacted with sodium hydroxide to form the yellow sodium salt. Residual diesel fuel is removed by ether extraction. The color is measured spectrophotometrically at 452 nm, and the concentration of amyl nitrate is determined by reference to a standard curve.

4. Significance and Use

4.1 In diesel fuel, the presence of alkyl nitrates such as amyl nitrate, hexyl nitrate, or octyl nitrate causes a higher residue value than observed in untreated fuel, which can lead to erroneous conclusions as to the coke forming propensity of the fuel. The presence of such alkyl nitrate in the fuel can be determined using this test method. This test method can be used to determine the amount of amyl nitrate that has been added to diesel fuels to improve cetane number. This test method is applicable as a basis for judging compliance with specifications covering amyl nitrate.

5. Interferences

5.1 Nitrate esters, inorganic nitrate ions, and nitrogen oxides will interfere to give high results.

6. Apparatus

6.1 *Absorption Cells*, matched, having a 1.000- \pm 0.002-cm light path.

6.2 *Distillate Collector*,³ borosilicate glass with standard taper joints and sealed-in funnel (Fig. 1).

6.3 *Electric Heating Mantle*, to fit a 300-mL round-bottom flask, and having a variable transformer.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products—Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The sole source of supply of the apparatus known to the committee at this time is obtainable from Corning Glass Co., Corning, NY, Catalog No. 3320. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

*A Summary of Changes section appears at the end of this standard

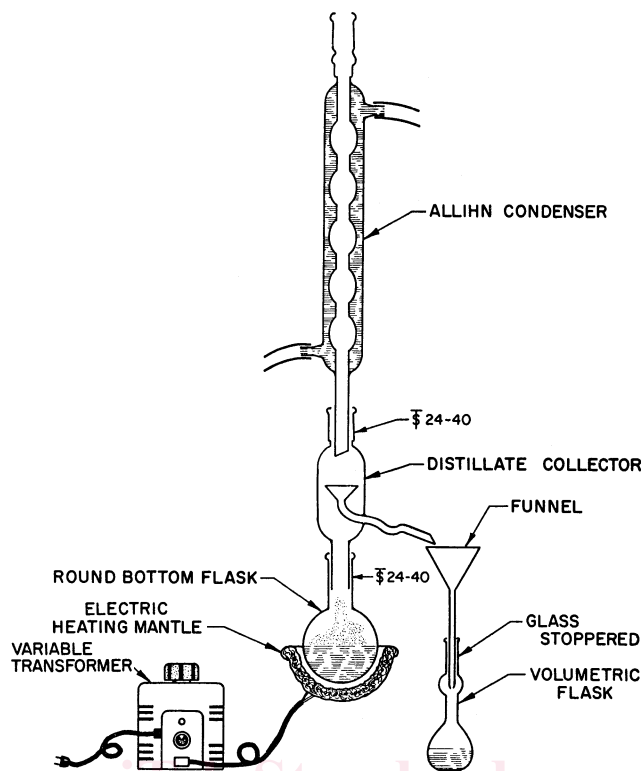


FIG. 1 Distillation Apparatus

6.4 Lunge-type Pipet, 2-mL capacity.

NOTE 2—A 2.5-mL hypodermic syringe, with scabbard, can be used instead of the Lunge pipet.

6.5 Reflux Condenser (Allihn-type), borosilicate glass, 300 mm—300 mm long, with standard-taper joints.

6.6 Round-Bottom Flask, of borosilicate glass and having a 300-mL capacity.

6.7 Separatory Funnel, Squibb-type, 125-mL capacity.

6.8 Shaking Machine, automatic, capable of 250 oscillations/min.

6.9 Spectrophotometer, capable of measuring absorption in the region of 452 nm.

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 shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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 specified, references to water shall be understood to mean reagent water as defined as Type II or III in Specification D1193.

7.3 Acetone (**Warning**—Extremely flammable. Vapors can cause flash fire. Harmful if inhaled.).

7.4 Amyl Nitrate—Any mixture of nitrate esters of primary amyl alcohol.

7.5 Diethyl Ether (**Warning**—Extremely flammable. Vapors can cause flash fire. Harmful if inhaled.).

7.6 Sodium Hydroxide Solution (20 g/L)—Dissolve 20 g of sodium hydroxide (NaOH) pellets in water and dilute to 1 L.

7.7 Sulfuric Acid (5 + 3)—Slowly add, with stirring, 500 mL—500 mL of nitrate-free concentrated sulfuric acid (H₂SO₄, rel dens 1.84) to 300 mL of water. The *m*-xylenol solution must be added before the H₂SO₄, otherwise low or negative results will be obtained. Cool before use.

⁴ 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.