



Designation: ~~D3237–06~~^{ε1} D3237–12

Standard Test Method for Lead in Gasoline by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D3237; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense.

^{ε1} NOTE—Corrected footnote references in Paragraph X1.6 editorially in April 2007.

1. Scope*

1.1 This test method covers the determination of the total lead content of gasoline within the concentration range of 0.010 to 0.10 g of lead/U.S. gal (2.5 to 25 mg/L). This test method compensates for variations in gasoline composition and is independent of lead alkyl type.

1.2 The values given in grams per U.S. gallon are to be regarded as the standard in the United States. Note that in other countries, other units can be preferred.

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 hazard statements, see 6.6 and 6.8.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[D1368 Test Method for Trace Concentrations of Lead in Primary Reference Fuels](#) (Withdrawn 1994)³ (Withdrawn 1994)³

[D2550 Method of Test for Water Separation Characteristics of Aviation Turbine Fuels](#) (Withdrawn 1989)³

[D3116 Test Method for Trace Amounts of Lead in Gasoline](#) (Withdrawn 1994)³ (Withdrawn 1994)³

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

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

[D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories](#)

[D7740 Practice for Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants](#)

3. Summary of Test Method

3.1 The gasoline sample is diluted with methyl isobutyl ketone and the alkyl lead components are stabilized by reaction with iodine and a quaternary ammonium salt. The lead content of the sample is determined by atomic absorption flame spectrometry at 283.3 nm, using standards prepared from reagent grade lead chloride. By the use of this treatment, all alkyl lead compounds give identical response.

3.2 Protocols for using atomic absorption spectrometry are given in Practice [D7740](#).

4. Significance and Use

4.1 This test method is used to ensure compliance of trace lead as required by federal regulation for lead-free gasoline (40 CFR part 80).

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 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 last approved version of this historical standard is referenced on www.astm.org.

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

5. Apparatus

- 5.1 *Atomic Absorption Spectrometer*, capable of scale expansion and nebulizer adjustment, and equipped with a slot burner and premix chamber for use with an air-acetylene flame.
- 5.2 *Volumetric Flasks*, 50-mL, 100-mL, 250-mL, and 1-L sizes.
- 5.3 *Pipets*, 2-mL, 5-mL, 10-mL, 20-mL, and 50-mL sizes.
- 5.4 *Micropipet*, 100- μ L, Eppendorf type or equivalent.

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.⁴

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Types II or III of Specification **D1193**.

6.3 *Aliquat 336* (tricapryl methyl ammonium chloride).

6.4 *Aliquat 336/MIBK Solution (10 % volume per volume)*—Dissolve and dilute 100 mL (88.0 g) of Aliquat 336 with MIBK to 1 L.

6.5 *Aliquat 336/MIBK Solution (1 % volume per volume)*—Dissolve and dilute 10 mL (8.8 g) of Aliquat 336 with MIBK to 1 L.

6.6 *Iodine Solution*—Dissolve and dilute 3.0 g of iodine crystals with toluene to 100 mL. (**Warning**—Flammable. Vapor harmful.)

6.7 *Lead Chloride* (PbCl_2).

6.8 *Lead-Sterile Gasoline*—Gasoline containing less than 0.005 g Pb/gal (1.32 mg Pb/L). (**Warning**—Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.)

NOTE 1—One way to confirm lead concentrations of less than 0.005 g Pb/gal (1.32 mg Pb/L) is to refer to Test Methods **D1368** and **D3116**. A procedure for the purification of gas turbine fuel appears in Appendix X4 of Test Method **D2550** and can be used to decrease the lead concentration of low lead gasoline in lead-sterile gasoline.

6.9 *Lead, Standard Solution (5.0 g Pb/gal (1.32 g Pb/L))*—Dissolve 0.4433 g of lead chloride (PbCl_2) previously dried at 105°C for 3 h in about 200 mL of 10 % Aliquat 336/MIBK solution in a 250-mL volumetric flask. Dilute to the mark with the 10 % Aliquat solution, mix, and store in a brown bottle having a polyethylene-lined cap. This solution contains 1321 μg Pb/mL, which is equivalent to 5.0 g Pb/gal.

6.10 *Lead, Standard Solution (1.0 g Pb/gal (264 mg Pb/L))*—By means of a pipet, accurately transfer 50.0 mL of the 5.0 g Pb/gal (1.32 g Pb/L) solution to a 250-mL volumetric flask, dilute to volume with 1 % Aliquat/MIBK solution. Store in a brown bottle having a polyethylene-lined cap.

6.11 *Lead, Standard Solutions (0.02, 0.05, and 0.10 g Pb/gal (5.3, 13.2, and 26.4 mg Pb/L))*—Transfer accurately by means of pipets 2.0, 5.0, and 10.0 mL of the 1.0-g Pb/gal (264 mg Pb/L) solution to 100-mL volumetric flasks; add 5.0 mL of 1 % Aliquat 336 solution to each flask; dilute to the mark with MIBK. Mix well and store in bottles having polyethylene-lined caps.

6.12 *Methyl Isobutyl Ketone (MIBK)*, (4-methyl-2-pentanone).

6.13 *Quality Control (QC) Samples*, preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section **11**.

7. Sampling

7.1 Take samples of gasoline in compliance with the instructions in Practice **D4057**.

7.2 Collect sample in a metal container that can be sealed against leakage and store under temperature-consistent conditions prior to analysis.

8. Calibration

8.1 *Preparation of Working Standards*—Prepare three working standards and a blank using the 0.02, 0.05, and 0.10-g Pb/gal (5.3, 13.2, and 26.4 mg Pb/L) standard lead solutions described in **6.11**.

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.