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An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

# Standard Test Method for Lead In Gasoline By Atomic Absorption Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation D 3237; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

### 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 Note 1 and Note 2.

#### 2. Referenced Documents

- 2.1 ASTM Standards:
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 1368 Test Method for Trace Concentrations of Lead in Primary Reference Fuels<sup>3</sup>
- D 2550 Test Method for Water Separation Characteristics of Aviation Turbine Fuels<sup>3</sup>
- D 3116 Test Method for Trace Amounts of Lead in Gasoline<sup>4</sup>
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>4</sup>

## 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.

### 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).

## 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-µL, 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.<sup>5</sup>
- 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 D 1193.
  - 6.3 Aliquat 336 6 (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.

Note 1—Warning: Flammable. Vapor harmful.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 05.02.

<sup>&</sup>lt;sup>5</sup> 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.

<sup>&</sup>lt;sup>6</sup> The sole source of supply of the solution known to the committee at this time is General Mills Corp., Minneapolis, MN 55415. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee<sup>1</sup>, which you may attend.