



Standard Test Method for Trace Amounts of Lead in Gasoline¹

This standard is issued under the fixed designation D 3116; 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. 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 from 0.001 g Pb/U.S. gal to 0.1 g Pb/U.S. gal (0.26 to 26 mg/L). The method was tested and found to be applicable to gasoline samples with olefin contents as high as 27 %.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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 3.*

1.3 The values stated in acceptable metric units are to be regarded as the standard. The values given in parentheses are for information purposes only.

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 1368 Test Method for Trace Concentrations of Lead in Primary Reference Fuels³
- D 3229 Test Method for Low Levels of Lead in Gasoline by Wavelength Dispersive X-Ray Spectrometry⁴
- D 3341 Test Method for Lead in Gasoline—Iodine Monochloride Method⁵
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵

3. Summary of Test Method

3.1 A known volume of sample is shaken with aqueous iodine monochloride solution which results in any lead compound present passing into the aqueous phase. Organo-lead compounds are then converted to the inorganic form by boiling the solution and the excess reagent is destroyed with sodium sulfite. After addition of buffer solution, the lead is extracted as the dithizonate and determined spectrophotometrically.

4. Significance and Use

4.1 This test method is used to determine trace levels of lead in unleaded gasolines. Unleaded gasoline is required for cars having catalytic converters. Lead will poison the catalyst in these converters.

5. Apparatus

5.1 All glassware must be borosilicate.

5.2 *Bottles*, borosilicate glass. All samples must be taken in borosilicate glass bottles.

5.3 *Flasks*, Erlenmeyer, 125-mL and 250-mL capacity.

5.4 *Separatory Funnels*, glass-stoppered and of sufficient capacity to accommodate the chosen sample size (for example, no more than four times the volume of sample plus reagents). Lubricate stopcocks with polytetrafluoroethylene (PTFE) aerosol spray or use PTFE stopcocks.

5.5 *Spectrophotometer*, capable of measuring light absorption at a wavelength of 520 nm.

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.⁶ 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 or III of Specification D 1193.

6.3 *Ammonium Citrate, Stock Solution (46 %)*—Weigh 200 g of citric acid monohydrate into a beaker, add about 50 mL of water and place the beaker in an ice-water cooling bath. Add sufficient *o*-cresol red indicator solution to give a definite color and then add the ammonia (relative density 0.880) slowly, with stirring, until the alkaline (red) color of the indicator is just developed. Dilute the solution to 500 mL with water and remove any lead present by extraction with successive portions of dithizone solution until the green color of the dithizone extract remains unchanged. Wash the solution with chloroform to remove any dissolved dithizone. Allow to stand until the aqueous phase is clear and separate

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.

Current edition approved March 31, 1989. Published June 1989. Originally published as D 3116 – 72. Last previous edition D 3116 – 82.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ Discontinued—See 1992 *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 05.02.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."