



## Standard Test Method for Lead in Gasoline—Iodine Monochloride Method<sup>1</sup>

This standard is issued under the fixed designation D 3341; 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.

### 1. Scope

1.1 This test method determines total lead in gasolines containing lead alkyls at concentrations between 0.026 and 1.3 g Pb/L, and 0.12 and 6.0 g Pb/UK gal, 0.1 and 5.0 g Pb/US gal.

1.2 The preferred units are grams per litre although both gram per US gallon and grams per UK gallon are acceptable due to their widespread use in the industry.

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 Sections 6 and 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance<sup>4</sup>

### 3. Summary of Test Method

3.1 A known volume of the sample is diluted with heavy distillate and shaken with aqueous iodine monochloride reagent. Any tetraalkyl lead compounds present react with the iodine monochloride and are extracted into the aqueous phase as the dialkyl lead compounds. The aqueous extract is separated from the gasoline and evaporated to low bulk to decompose free iodine monochloride. Any organic matter present is removed by oxidation with nitric acid, which also serves to convert the dialkyl lead compounds into inorganic lead compounds. The residue is dissolved in distilled water and buffered

to pH 5 using sodium acetate-acetic acid buffer. The lead content of the buffered solution is determined by titration with EDTA using xylenol orange as indicator.

### 4. Significance and Use

4.1 This test method determines the concentration of lead alkyl additives in gasoline. These additives improve the anti-knock properties.

### 5. Apparatus

5.1 *Separatory Funnel*, borosilicate glass, capacity 250 mL, glass-stoppered with preferably an iodine flask type of neck.

5.2 *Erlenmeyer Flask*, borosilicate glass, capacity 500 mL.

### 6. Reagents and Materials

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> 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 Type III or Type IV of Specification D 1193.

6.3 *Ammonia Solution (1 + 1)*—Mix 1 volume of concentrated ammonia solution (rel dens 0.90) with 1 volume of distilled water.

6.4 *Bromthymol Blue Indicator Solution*—Dissolve 0.1 g of bromthymol blue in 50 mL of ethanol and dilute to 100 mL with distilled water.

6.5 *EDTA, Standard Solution (0.005 M)*—Dissolve approximately 3.75 g of diaminoethanetetra-acetic acid, disodium salt, in 2-L of distilled water. Determine the molarity of the solution by standardization with lead nitrate solution as follows:

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.03.

<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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.