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An American National Standard

Standard Test Method for Trace Concentrations of Lead in Primary Reference Fuels¹

This standard is issued under the fixed designation D 1368; 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 trace quantities of lead in primary reference fuels contaminated with trace amounts of alkyl lead antiknock compounds or other lead compounds. The method is specified for the range 0.001 to 0.003 g of lead per U.S. gallon (0.26 to 0.79 mg/L), the concentration of significance for contaminated primary reference fuels.

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 Notes 1 through 7.*

1.3 The values in acceptable metric units are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

3. Summary of Test Method

3.1 The sample is treated with bromine, heated on a steam bath to decompose the alkyl lead and alkyl lead salts, and then extracted with dilute nitric acid. The pH of the aqueous extract is adjusted by means of a buffer and the lead is extracted with a chloroform solution of dithizone. The absorbance of the chloroform extract is measured and the lead content is determined from a previously prepared calibration curve.

4. Significance and Use

4.1 Knowledge of the concentration of lead contamination of primary reference fuels is necessary if the fuel is to be used as a reference fuel for engine testing.

5. Apparatus

5.1 *Borosilicate Glassware* shall be used in this method.

5.2 *Spectrophotometer*, fitted with covered absorption cells having a 1-cm light path.

5.3 *Shaking Machine* (optional) capable of approximately 250 cpm.

5.4 *Separatory Funnels*, Squibb-type, 125-mL volume.

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 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 *Bromine Solution*—Dilute 300 mL of bromine (Warning—see Note 1) to 1000 mL with carbon tetrachloride (Warning—see Note 2). Filter through a sintered-glass filter before using.

NOTE 1: Warning—Corrosive liquid.

NOTE 2: Warning—Vapor harmful. Poison. Harmful to eyes or fatal if swallowed. Absorbed through the skin. Known to cause liver and kidney damage. Will produce toxic vapors if burned.

NOTE 3: Precaution—In addition to other precautions, handle bromide and bromine solutions in vented hood. Neutralize spills with soda ash or alkaline solution.

6.4 *Buffer Solution*—Dissolve 20 g of potassium cyanide (KCN) (Warning—see Note 4), 6 g of ammonium citrate, and 6 g of sodium sulfite (Na_2SO_3) separately in water. Mix the solutions, add 150 mL of concentrated ammonium hydroxide (NH_4OH , relative density 0.90), and dilute to 1000 mL with water. This solution is stable for only 3 months.

NOTE 4: Warning—Poisonous solid.

NOTE 5: Precaution—In addition to other precautions, handle KCN with rubber gloves in vented hood. Drain excess and waste solution in a hooded sink not used for the disposal of acids, and flush with water.

6.4.1 Store the solutions described in 6.4 and 6.5 in the refrigerator, but bring them to room temperature before using.

6.5 *Dithizone Solution*—Dissolve 30 mg of dithizone⁵ in 1000 mL of chloroform. This solution is stable for only 4 weeks.

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.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 05.03.

⁴ "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."

⁵ Eastman Kodak white label dithizone has been found satisfactory for this purpose.