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An American National Standard

H-24-15

Standard Test Method for LOW LEVELS OF LEAD IN GASOLINE BY X-RAY SPECTROMETRY¹

This standard is issued under the fixed designation D 3229; 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 total lead in gasoline in the range from 0.010 to 0.50 g Pb/U.S. gal.

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see 6.3, 6.4, 6.5, and 6.7.

2. Applicable Documents

2.1 ASTM Standards:

D2599 Test Method for Lead in Gasoline by X-Ray Spectrometry²

D3341 Test Method for Lead in Gasoline—Iodine Monochloride Method³

3. Summary of Method

3.1 Twenty millilitres of sample is mixed thoroughly with 2 mL of bismuth internal standard solution. The mixture is placed in the X-ray beam of a spectrometer and the intensities of the lead $L\alpha_1$ radiation at 1.175 Å, the bismuth $L\alpha_1$ radiation at 1.144 Å, and a background at 1.194 Å are determined. A blank, made with isooctane and bismuth internal standard, is run using the same procedure. The lead concentration is measured by determining the ratio of the net counting rate at 1.175 Å to the gross counting rate at 1.144 Å for the sample, subtracting the comparable ratio found for the blank, and comparing to a previously prepared calibration curve of concentration versus the same ratios.

4. Significance and Use

4.1 This test method is used to ensure com-

pliance of trace lead as required by federal regulations for lead-free gasoline (40 CFR part 80).

5. Apparatus

5.1 *X-ray Spectrometer*, capable of measuring radiations mentioned in 3.1 and of being operated under the following instrumental conditions or others giving equivalent results:

Tube voltage—50 kV
Tube current—45 mA
Analyzing crystal—lithium fluoride (LiF)
Optical path—air, helium, or hydrogen
Detector—proportional or scintillation

NOTE 1: **Caution**—The X-ray spectrometer and manner of use should comply with the regulations covering the use of ionizing radiation or recommendations of the International Commission on Radiological Protection, or both.

Where hydrogen is used in the optical path, there is a potential explosion hazard.

6. Reagents and Materials

6.1 Bismuth 2-Ethylhexoate⁴

6.2 *Bismuth 2-Ethylhexoate Internal Standard Solution*—Dilute the bismuth 2-ethylhexoate with heavy, odorless solvent or similar material to obtain a solution containing 3.00 g Bi/U.S. gal at 60°F (15.6°C).

NOTE 2—Some stability difficulties have been experienced with the bismuth internal standard solution. If the standard is blended to contain 5 % 2-ethylhexanoic acid the standard has been found to last almost indefinitely. The 2-ethylhexanoic acid stabilizes isooct-

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 Analyses.

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² Annual Book of ASTM Standards, Vol 05.02.

³ Annual Book of ASTM Standards, Vol 05.03.

⁴ Available from Angstrom, Inc., P.O. Box 252, Belleville, MI 48111.