



Standard Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry¹

This standard is issued under the fixed designation D3120; 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.

1. Scope*

1.1 This test method covers the determination of sulfur concentration in the range from 3.0 to 1000 mg/kg in light liquid hydrocarbons and fuels with oxygenates boiling in the range from 26 to 274°C (80 to 525°F).

NOTE 1—Preliminary data has shown that this test method is also applicable to the determination of sulfur in denatured fuel ethanol (Specification D4806), automotive spark ignition engine fuel (Specification D4814), Ed75–Ed85 (Specification D5798) or gasoline-oxygenate fuel blends with greater than 10% ethanol. However, the precision for these materials has not been determined. Subcommittee D02.03 is undertaking activities to obtain precision statements for these materials.

1.2 Other materials falling within the distillation range specified in 1.1, but having sulfur concentrations above 1000 mg/kg, may be tested using appropriate dilutions to bring them within the specified limit. In addition, sample types that may be outside the specified distillation range, such as diesels and biodiesels, may be analyzed by this test method.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. The preferred units are milligrams per kilogram (mg/kg).

1.4 *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 7-9.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

¹ 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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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel

D4814 Specification for Automotive Spark-Ignition Engine Fuel

D5798 Specification for Fuel Ethanol (Ed75–Ed85) for Automotive Spark-Ignition Engines

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

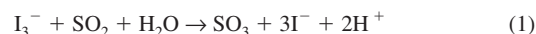
2.2 OSHA Regulations:

OSHA Regulations 29 CFR, paragraphs 1910.1000 and 1910.1200³

3. Summary of Test Method

3.1 A liquid sample is introduced into a pyrolysis tube maintained at a temperature between 900–1200°C, having a flowing stream of gas containing 50–80% oxygen and 20–50% inert gas (for example, argon, helium, etc.) Oxidative pyrolysis converts the sulfur to sulfur dioxide, which then flows into a titration cell where it reacts with triiodide ion present in the electrolyte. The triiodide ion consumed is coulometrically replaced and the total current ($I \times t$) required to replace it is a measure of the sulfur present in the sample.

3.2 The reaction occurring in the titration cell as sulfur dioxide enters is:



The triiodide ion consumed in the above reaction is generated coulometrically thus:

³ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

*A Summary of Changes section appears at the end of this standard.