



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

This standard is issued under the fixed designation D 3120; 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 boiling in the range from 26 to 274°C (80 to 525°F).

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 the standard. The values given in parentheses are provided for information only. 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:

D 1193 Specification for Reagent Water²

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method³

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

D 4177 Practice for Automatic Sampling of Petroleum and

Petroleum Products⁴

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁵

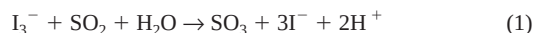
2.2 OSHA Regulations:

OSHA Regulations 29 CFR, paragraphs 1910.1000 and 1910.1200⁶

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:



3.3 These microequivalents of triiodide ion (iodine) are equal to the number of microequivalents of titratable SO_2 ion entering the titration cell.

4. Significance and Use

4.1 This test method is used to determine the concentration of sulfur in light liquid hydrocarbons, gasoline, and diesels and their additives, where such concentrations of sulfur can be detrimental to their production, performance, and use. The measurement of sulfur in the production and final product of gasoline and diesel is required for both regulatory purposes and to ensure maximum life expectancy of catalytic converters used in the automotive industry.

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 Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 05.02.

⁵ Annual Book of ASTM Standards, Vol 05.03.

⁶ 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.

