



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

This standard is issued under the fixed designation D 3961; 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 determines total sulfur content in aromatic hydrocarbons, their derivatives, and related chemicals.

1.2 This test method is applicable to samples with sulfur concentrations from 0.5 to 100 mg/kg.

1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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 Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3437 Practice for Sampling and Handling Liquid Cyclic Products³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

2.2 Other Document:

OSHA Regulations - 29 CFR paragraphs 1910.1000 and 1910.1200⁵

3. Terminology

3.1 Definitions:

3.1.1 *dehydration tube*—a chamber containing phosphoric acid 85 % that scrubs the effluent gases from combustion to remove water vapor.

3.1.2 *oxidative pyrolysis*—a process in which a sample is combusted in an oxygen-rich atmosphere at high temperature to break down the components of the sample into elemental oxides.

3.1.3 *recovery factor*—an indication of the efficiency of the measurement computed by dividing the measured value of a standard by its theoretical value.

3.1.4 *reference sensor pair*—detects changes in triiodide ion concentrations.

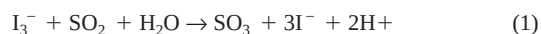
3.1.5 *titration parameters*—various instrumental conditions that can be changed for different types of analysis.

3.1.6 *working electrode (generator electrode)*—an electrode consisting of an anode and a cathode separated by a salt bridge or a liquid electrolyte bridge; maintains a constant triiodide ion concentrations.

4. Summary of Test Method

4.1 A liquid specimen is injected into a combustion tube that can be maintained between 900° to 1200° C and having a flowing stream of gas containing approximately 50 to 75 % oxygen and 25 to 50 % inert gas (for example helium, argon, 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 thus consumed is coulometrically replaced and the total electrical work required to replace it is a measure of the sulfur present in the sample injected.

4.2 The reaction is generated in the titration cell as sulfur dioxide as follows:



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



¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

Current edition approved May 10, 1998. Published August 1998. Originally published as D 3961 – 80. Last previous edition D 3961 – 89 (1993).^{ε1}

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 06.04.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.