



Test Method for Total Sulfur in Liquid Aromatic Hydrocarbons and Their Derivatives by Oxidative Combustion and Electrochemical Detection¹

This standard is issued under the fixed designation D 6428; 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 the total sulfur naturally found in liquid aromatic hydrocarbons, their derivatives and related chemicals.

1.2 This test method is applicable for samples containing sulfur from 0.05 to 100 mgS/kg.

1.3 The detector response for this technique within the scope of this test method is linear with sulfur concentration.

1.4 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.

1.5 *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, and Notes 2-4 and Note 7.

2. Referenced Documents

2.1 ASTM Standards:

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

D 3852 Practice for Sampling and Handling Phenol and Cresylic Acid

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

2.2 Other Documents

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁴

3. Terminology

3.1 Definitions:

3.1.1 *oxidative combustion, n*—process in which a sample under goes combustion in an oxygen rich environment at temperatures greater than of 650°C and compounds decompose to carbon dioxide, water, and elemental oxides.

3.1.2 *quartz combustion tube devitrification, n*—the suggested maximum temperature for a quartz combustion tube is 1200°C.

3.1.2.1 *Discussion*—Samples containing alkali-metals (elements from the Periodic Group IA (that is, sodium, potassium, etc.)) or alkaline earth (elements from the Periodic Group IIA (that is, calcium, magnesium, etc.)) will cause quartz to devitrify (that is, become milky white and brittle).

4. Summary of Test Method

4.1 The sample of liquid aromatic hydrocarbon is injected, at a controlled rate, into a stream of inert gas (helium or argon) or inert gas mixed with oxygen. The sample is vaporized and carried into a high temperature zone (>900°C) where oxygen is introduced. Sulfur compounds, present in the specimen are converted to sulfur dioxide (SO₂). Sulfur dioxide is reacted with the sensing electrode in a 3-electrode electrochemical cell. This reaction produces a measurable current that is directly proportional to the amount of sulfur in the original sample material. The reaction that occurs is as follows:

Combustion: $R-S + O_2 \xrightarrow{900^\circ C} CO_2 + H_2O + SO_2 + \text{oxides}$
Detection: $SO_2 + 2 H_2O \xrightarrow{\text{electrolyte}} H_2SO_4 + (2H^+) + (2e^-)$

5. Significance and Use

5.1 Some process catalysts used in petroleum and chemical refining may be poisoned when even trace amounts of sulfurous materials are contained in the feedstocks. This test method can be used to determine total sulfur in process feeds and may also be used to control sulfur compounds in finished products which fall within the scope of this test method.

NOTE 1—Virtually all sulfur compounds will be detected by this technique.

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.

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² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.