



Standard Test Method for Total Sulfur in Aromatic Compounds by Hydrogenolysis and Rateometric Colorimetry¹

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1. Scope

1.1 This test method covers the determination of sulfur in aromatic hydrocarbons, their derivatives, and related chemicals having typical sulfur concentrations from 0.020 to 10 mg/kg.

1.2 This test method may be extended to higher concentrations by dilution.

1.3 This test method is applicable to aromatic hydrocarbons and related chemicals such as benzene, toluene, cumene, *p*-xylene, *o*-xylene, and to cyclohexane.

1.4 The following applies to all specified limits in this test method: for purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded off to the nearest unit in the last right-hand digit used for 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 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.* Specific precautionary statements are given in 6.4, 7.5, 7.7, and 8.1.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3437 Practice for Sampling and Handling Liquid Cyclic Products³

D 4045 Test Method for Sulfur in Petroleum Products by Hydrogenolysis and Rateometric Colorimetry⁴

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

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals³

E 29 Practice for Using Significant Digits in Test Digits in

Test Data to Determine Conformance with Specifications⁵

2.2 Other Documents:

OSHA Regulations, 29 CFR paragraphs 1910.1 and 1910.1200⁶

3. Terminology

3.1 See Terminology D 4790 for definition of terms used in this test method.

4. Summary of Test Method

4.1 *Reductive Configuration*—The sample is injected at a constant rate into a hydrogenolysis apparatus. Within this apparatus the sample is pyrolyzed at *temperatures in the range of 1200°C to 1300°C* and in the presence of excess hydrogen. Sulfur compounds are reduced to hydrogen sulfide (H₂S). Analysis is by rateometric detection of the colorimetric reaction of H₂S with lead acetate. Hydrocarbon components are converted to gaseous such as methane during hydrogenolysis.

4.2 *OxyhydroPyrolysis Configuration*—Sample is injected at a constant rate into an air stream and introduced into a pyrolysis furnace. The sample flows through an inner tube within the furnace where it combusts with the oxygen in the air carrier. SO₂ and SO₃ are formed from the sulfur compounds in the sample. The sample then leaves the inner tube within the pyrolyzer and is mixed with hydrogen within the main reaction tube and is pyrolyzed at *temperatures in the range of 1200°C to 1300°C* (see Fig. 1). The SO₂ and SO₃ formed within the inner tube are then reduced to H₂S. Analysis is by rateometric detection of the colorimetric reaction of H₂S with lead acetate.

5. Significance and Use

5.1 Sulfur can be a catalyst poison in the aromatic chemical manufacturing process. This test method can be used to monitor the amount of sulfur in aromatic hydrocarbons. This test method may also be used as a quality control tool and in setting specifications for sulfur determination in finished products.

6. Apparatus

6.1 The apparatus of this test method can be setup in two

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 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.