



Designation: D 4735 – 96

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determination of Trace Thiophene in Refined Benzene by Gas Chromatography¹

This standard is issued under the fixed designation D 4735; 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 thiophene in refined benzene in the range from 0.5 mg/kg to 5.0 mg/kg. The range of the test method may be extended by modifying the sample injection volume, calibration range, or sample dilution with thiophene-free solvent.

1.2 This test method has been found applicable to benzene characteristic of the type described in Specifications D 2359 and D 4734 and may be applicable to other types or grades of benzene only after the user has demonstrated that the procedure can completely resolve thiophene from the other organic contaminants contained in the sample.

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 7.

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 1685 Test Method for Traces of Thiophene in Benzene by Spectrophotometry³
- D 2359 Specification for Refined Benzene-535³
- D 3437 Practice for Sampling and Handling Liquid Cyclic Products³
- D 4734 Specification for Refined Benzene 545³
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

¹ This test method is under the jurisdiction of ASTM Committee D-16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.0E 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 14.02.

E 260 Practice for Packed Column Gas Chromatography⁴

E 840 Practice for Using Flame Photometric Detectors in Gas Chromatography⁴

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁵

3. Summary of Test Method

3.1 The thiophene concentration in refined benzene is determined at the milligram thiophene per kilogram sample level using conventional gas-liquid chromatography with a flame photometric detector. A reproducible volume of sample is injected. Quantitative results are obtained by the external standard technique using the measured peak area of thiophene.

4. Significance and Use

4.1 This test method is suitable for setting specifications on benzene and for use as an internal quality control tool where benzene is either produced or used in a manufacturing process.

4.2 This test method was found applicable for determining thiophene in refined benzene conforming to the specifications described in Specification D 2359 and may be applicable toward other grades of benzene if the user has taken the necessary precautions as described in the text.

4.3 This test method was developed as an alternative technique to Test Method D 1685.

5. Apparatus

5.1 *Gas Chromatograph*—Any chromatograph having a flame photometric detector may be used which can operate at the typical conditions described in Table 1. The user is referred to Practice E 260 for additional information about gas chromatography principles and procedures.

5.2 *Column*—The column must provide complete resolution of thiophene from benzene and any other hydrocarbon impurities because of potential quenching effects by hydrocarbons on the light emissions from the thiophene. The columns described in Table 1 have been judged satisfactory.

5.3 *Detector*—Any flame photometric detector (FPD) can be used, provided it has sufficient sensitivity to produce a minimum peak height twice that of the base noise for a 4-μL

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