



Designation: D 5441 – 93

Standard Test Method for Analysis of Methyl Tert-Butyl Ether (MTBE) by Gas Chromatography¹

This standard is issued under the fixed designation D 5441; 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 provides for determination of the purity of methyl tert-butyl ether (MTBE) by gas chromatography. It also provides a procedure to measure impurities in MTBE such as C₄ to C₁₂ olefins, methyl, isopropyl and tert-butyl alcohols, methyl sec-butyl and methyl tert-amyl ethers, acetone, and methyl ethyl ketone. Impurities are determined to a minimum concentration of 0.02 mass %.

1.2 This test method is not applicable to the determination of MTBE in gasoline.

1.3 Water cannot be determined by this test method and must be measured by a procedure such as Test Method D 1364 and the result used to normalize the chromatographic values.

1.4 A majority of the impurities in MTBE is resolved by the test method, however, some co-elution is encountered.

1.5 This test method is inappropriate for impurities that boil at temperatures higher than 180°C or for impurities that cause poor or no response in a flame ionization detector, such as water.

1.6 The values stated in SI (metric) units of measurement are preferred and used throughout the standard. Alternate units, in common usage, are also provided to improve clarity and aid the user of this method.

1.7 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1364 Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)²
- D 3700 Practice for Containing Hydrocarbon Fluid Samples Using Floating Piston Cylinder³
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³
- D 4307 Practice for Preparation of Liquid Blends for Use as

Analytical Standards³

- D 4626 Practice for Calculation of Gas Chromatographic Response Factors³
- E 355 Practice for Gas Chromatography Terms and Relationships⁴
- E 594 Practice for Testing Flame Ionization Detectors Used in Gas Chromatography⁴

3. Terminology

3.1 Definitions:

3.1.1 This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions of these can be found in Practices E 355 and E 594.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 C₄ to C₁₂ olefins— common olefin impurities in MTBE are unreacted feedstock and dimers or trimers of feed such as trimethylpentene or pentamethylheptene.

4. Summary of Test Method

4.1 A representative aliquot of the MTBE product sample is introduced into a gas chromatograph equipped with a methyl silicone bonded phase fused silica open tubular column. Helium carrier gas transports the vaporized aliquot through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column.

4.2 The detector signal is processed by an electronic data acquisition system or integrating computer. Each eluting component is identified by comparing its retention time to those established by analyzing standards under identical conditions.

4.3 The concentration of each component in mass percent is determined by normalization of the peak areas after each peak area has been corrected by a detector response multiplication factor. The detector response factors are determined by analyzing prepared standards with concentrations similar to those encountered in the sample.

5. Significance and Use

5.1 The presence of impurities in MTBE product can have a deleterious effect upon the value of MTBE as a gasoline

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.D0.05 on C₅ Hydrocarbons and Oxygenated Hydrocarbons.

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² Annual Book of ASTM Standards, Vol 06.04.

³ Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 14.01.