



Standard Test Method for Determination of Trace Oxygenates in Automotive Spark-Ignition Engine Fuel by Multidimensional Gas Chromatography¹

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1. Scope*

1.1 This test method covers the determination of trace oxygenates in automotive spark-ignition engine fuel. The method used is a multidimensional gas chromatographic method using 1,2-dimethoxy ethane as the internal standard. The oxygenates that are analyzed are: methyl-tertiary butyl ether (MTBE), ethyl-tertiary butyl ether (ETBE), diisopropyl ether (DIPE), methanol, tertiary-amyl methyl ether (TAME), n-propanol, i-propanol, n-butanol, i-butanol, tert-butyl alcohol, sec-butyl alcohol, and tert-pentanol. Ethanol is usually not measured as a trace oxygenate since ethanol can be used as the main oxygenate compound in finished automotive spark-ignition fuels such as reformulated automotive spark-ignition fuels. The concentration range of the oxygenates covered in the ILS study was from 10 mg/kg to 2000 mg/kg. In addition this method is also suitable for the measurement of the C5 isomeric alcohols (2-methyl-1-butanol, 2-methyl-2-butanol) present from the fermentation of ethanol.

1.2 The ethanol blending concentration for which this test method applies ranges from 1 % to 15% by volume. Higher concentrations of ethanol coelute with methanol in the analytical column. Lower levels of ethanol, similar to the other oxygenate, can be calibrated and analyzed also. If higher ethanol concentrations are expected, the window cutting technique can be used to avoid ethanol from entering the analytical column and interfere with the determination of the other oxygenates of interest. Refer to [Appendix X1](#) for details.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 Alternative units, in common usage, are also provided to increase clarity and aid the users of this test method.

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.

2. Referenced Documents

2.1 ASTM Standards:²

- [D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)
- [D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards](#)
- [D4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C₁ to C₄ Alcohols in Gasoline by Gas Chromatography](#)
- [D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration](#)

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *electronic pressure control*, *n*—electronic pneumatic control of carrier gas flows. Can be flow or pressure programmed to speed up elution of components.

3.1.2 *flame ionization detector (FID)*, *n*—detector used to analyze the components eluting from the column.

3.1.3 *fluidic switch*, *n*—device that reverses the directional flow in a union T altering the pressure at the midpoint. In its simplest design it is also known as a Dean Switch.

3.1.4 *inlet*, *n*—capillary split/splitless inlet system operated in the split mode is recommended. Operate the inlet within its linear range.

3.1.4.1 *split ratio*, *n*—*in capillary gas chromatography*, the ratio of the total flow of carrier gas to the sample inlet versus the flow of the carrier gas to the capillary column is expressed by:

$$\text{Split ratio} = (S + C)/C \quad (1)$$

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.