



Standard Test Method for Determination of Olefin Content in Denatured Ethanol by Supercritical Fluid Chromatography¹

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1. Scope*

1.1 This test method covers the determination of the total amount of olefins in denatured ethanol to be used as an oxygenate additive in blended spark ignition engine fuels. The method of determination is supercritical fluid chromatography (SFC). The application range is from 0.1 mass percent to 1.0 mass percent total olefins. Results are expressed in terms of mass percent olefins.

1.2 This test method can be used for the analysis of denatured ethanol that is intended to be used as an oxygenate additive in commercial spark ignition engine fuels.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D5186 Test Method for Determination of the Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels By Supercritical Fluid Chromatography

D6550 Test Method for Determination of Olefin Content of Gasolines by Supercritical-Fluid Chromatography

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 D02.04.0C on Liquid Chromatography.

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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.

3. Terminology

3.1 Definitions:

3.1.1 *critical pressure, n*—that pressure needed to condense a gas at the critical temperature.

3.1.2 *critical temperature, n*—highest temperature at which a gaseous fluid can be converted to a liquid by means of compression.

3.1.3 *supercritical fluid, n*—fluid maintained in a thermodynamic state above its critical temperature and critical pressure.

3.1.4 *supercritical fluid chromatography, n*— class of chromatography that employs supercritical fluids as mobile phases.

4. Summary of Test Method

4.1 A small aliquot of the denatured alcohol sample is injected onto a set of three analytical chromatographic columns connected in series. The sample is transported through the columns using supercritical carbon dioxide (CO₂) as the mobile phase. The first column is packed with polyvinyl alcohol (PVA). The second column in the series is an analytical column packed with high surface area silica gel particles, and the third column is packed with silica particles coated with strong cation exchange material loaded with silver ions.

4.2 Two six-port switching valves are used to direct the different classes of components through the chromatographic system to the detector. In a forward flow mode, saturates, aromatics, and olefins pass onto the analytical silica gel column while the alcohol is retained on the PVA column. The saturates, aromatics, and olefins are maintained on the silica column, while the alcohol is back-flushed to the detector. This step frees the flow path of alcohol species allowing for the separation of the olefins from saturates and aromatics. The forward flow mode is resumed after the alcohol is eliminated and saturates are carried to the detector, while the aromatics are retained on the silica column and the olefinic species are trapped on the silver-loaded column. The next step is to back-flush the olefins from the silver-loaded column to the detector. Finally the aromatics are carried from the silica column to the detector in a forward flow mode, bypassing the silver-loaded column.

4.3 A flame ionization detector (FID) is used for quantitation. Calibration is based on the area of the chromatographic

*A Summary of Changes section appears at the end of this standard