Designation: D7059 – 09 (Reapproved 2013)

Standard Test Method for Determination of Methanol in Crude Oils by Multidimensional Gas Chromatography[1]

This standard is issued under the fixed designation D7059; 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 methanol in crude oils by direct injection multidimensional gas chromatography in the concentration range of 15 to 900 ppm (m/m). The pooled limit of quantification (PLOQ) is 15 ppm (m/m).

1.2 This test method is applicable only to crude oils containing less than or equal to 0.1 % (v/v) water.

1.3 This test method has not been tested with crude oil samples that are solid or waxy, or both, at ambient temperatures.

1.4 The values stated in SI units are to be regarded as standard. Alternate units, in common usage, are also provided to increase clarity and aid the users of this test method.

1.5 *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]

D4006 Test Method for Water in Crude Oil by Distillation

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration

D6596 Practice for Ampulization and Storage of Gasoline and Related Hydrocarbon Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *analytical column, n*—porous layer open tubular (PLOT) column with a stationary phase selective for oxygenates. It is used to resolve methanol from 1-propanol to provide accurate quantitative results.

3.1.2 *cool-on-column injector, n*—an injection port that allows controlled injection of the sample at a temperature close to or lower than the boiling point of the solvent into the gas chromatographic column or a liner within the injection port connected to the column.

3.1.2.1 *Discussion*—After the injection, the injection port is heated at a fixed rate to a temperature sufficiently high enough to allow the transfer of sample components of interest from the injection port to the part of the column located in the gas chromatograph (GC) oven.

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

3.1.4 *low-volume connector, n*—a special union for connecting two lengths of tubing 1.6-mm inside diameter and smaller; sometimes referred to as a zero dead-volume union.

3.1.5 *pre-column, n*—a polydimethylsiloxane WCOT column used to isolate the methanol and 1-propanol and several light hydrocarbons from the higher boiling portion of the crude oil sample for transfer to the analytical column for further separation and quantification.

3.1.6 *programmable temperature vaporizer (PTV), n*—a temperature programmable injector similar to a cool-on-column injector except that the sample is injected cool into a glass liner or insert instead of the WCOT (3.1.5) column and then the temperature is programmed in a manner similar to the on-column injector.

3.1.6.1 *Discussion*—The liner may be replaced, as necessary, to remove non-volatile materials. This injector may be operated in low split mode or direct (no splitting) mode.

3.1.7 *split/splitless injector, n*—a heated capillary inlet or sample introduction system that allows controlled splitting of the injected sample into two unequal portions, the smaller of which goes to the capillary column, and the greater to a vent.

3.1.7.1 *Discussion*—When the vent is closed, the entire

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand 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.

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