



Designation: D6730 – 22

Standard Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100-Metre Capillary (with Precolumn) High- Resolution Gas Chromatography¹

This standard is issued under the fixed designation D6730; 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 individual hydrocarbon components of spark-ignition engine fuels and their mixtures containing oxygenate blends (MTBE, ETBE, ethanol, and so forth) with boiling ranges up to 225 °C. Other light liquid hydrocarbon mixtures typically encountered in petroleum refining operations, such as blending stocks (naphthas, reformates, alkylates, and so forth) may also be analyzed; however, statistical data was obtained only with blended spark-ignition engine fuels.

1.2 Based on the cooperative study results, individual component concentrations and precision are determined in the range from 0.01 % to approximately 30 % by mass. The test method may be applicable to higher and lower concentrations for the individual components; however, the user must verify the accuracy if the test method is used for components with concentrations outside the specified ranges.

1.3 This test method also determines methanol, ethanol, *t*-butanol, methyl *t*-butyl ether (MTBE), ethyl *t*-butyl ether (ETBE), and *t*-amyl methyl ether (TAME) in spark ignition engine fuels in the concentration range from 1 % to 30 % by mass. However, the cooperative study data provided insufficient statistical data for obtaining a precision statement for these compounds.

1.4 Although a majority of the individual hydrocarbons present are determined, some co-elution of compounds is encountered. If this test method is utilized to estimate bulk hydrocarbon group-type composition (PONA), the user of such data should be cautioned that some error will be encountered due to co-elution and a lack of identification of all components present. Samples containing significant amounts of naphthenic (for example, virgin naphthas) constituents above *n*-octane may reflect significant errors in PONA-type groupings. Based

on the gasoline samples in the interlaboratory cooperative study, this test method is applicable to samples containing less than 25 % by mass of olefins. However, some interfering co-elution with the olefins above C_7 is possible, particularly if blending components or their higher boiling cuts such as those derived from fluid catalytic cracking (FCC) are analyzed, and the total olefin content may not be accurate. **Annex A1** of this test method compares results of the test method with other test methods for selected components, including olefins, and several group types for several interlaboratory cooperative study samples. Although benzene, toluene, and several oxygenates are determined, when doubtful as to the analytical results of these components, confirmatory analyses can be obtained by using the specific test methods listed in the reference section.

1.4.1 Total olefins in the samples may be obtained or confirmed, or both, if necessary, by Test Method **D1319** (percent by volume) or other test methods, such as those based on multidimensional PONA-type of instruments.

1.5 If water is or is suspected of being present, its concentration may be determined, if desired, by the use of Test Method **D1744** or equivalent. Other compounds containing oxygen, sulfur, nitrogen, and so forth, may also be present, and may co-elute with the hydrocarbons. If determination of these specific compounds is required, it is recommended that test methods for these specific materials be used, such as Test Methods **D4815** and **D5599** for oxygenates, and Test Method **D5623** for sulfur compounds, or equivalent.

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

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

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 **D02.04.01** on Gas Chromatography Methods.

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

Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

- D1319** Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption
- D1744** Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent (Withdrawn 2016)³
- D3700** Practice for Obtaining LPG Samples Using a Floating Piston Cylinder
- D4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D4177** Practice for Automatic Sampling of Petroleum and Petroleum Products
- D4307** Practice for Preparation of Liquid Blends for Use as Analytical Standards
- D4626** Practice for Calculation of Gas Chromatographic Response Factors
- D4815** Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C₁ to C₄ Alcohols in Gasoline by Gas Chromatography
- D5580** Test Method for Determination of Benzene, Toluene, Ethylbenzene, *p/m*-Xylene, *o*-Xylene, C₉ and Heavier Aromatics, and Total Aromatics in Finished Gasoline by Gas Chromatography
- D5599** Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection
- D5623** Test Method for Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection
- E355** Practice for Gas Chromatography Terms and Relationships
- E594** Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography
- E1510** Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

3. Terminology

3.1 Definitions—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practice **E355**.

4. Summary of Test Method

4.1 A representative sample of the petroleum liquid is introduced into a gas chromatograph equipped with an open tubular (capillary) column coated with a methyl silicone liquid phase, modified with a capillary precolumn. Helium carrier gas transports the vaporized sample through the column, in which

it is partitioned into individual components which are sensed with a flame ionization detector as they elute from the end of the column. The detector signal is presented on a strip chart recorder or digitally, or both, by way of an integrator or integrating computer. Each eluting component is identified by comparing its retention time to that established by analyzing reference standards or samples under identical conditions. The concentration of each component in percent by mass is determined by normalization of the peak areas after correction with detector response factors. Unknown components are reported as a total unknown percent by mass.

5. Significance and Use

5.1 Knowledge of the individual component composition (speciation) of gasoline fuels and blending stocks is useful for refinery quality control and product specification. Process control and product specification compliance for many individual hydrocarbons can be determined through the use of this test method.

5.2 This test method is adopted from earlier development and enhancement.^{4,5,6,7} The chromatographic operating conditions and column tuning process, included in this test method, were developed to provide and enhance the separation and subsequent determination of many individual components not obtained with previous single-column analyses. The column temperature program profile is selected to afford the maximum resolution of possible co-eluting components, especially where these are of two different compound types (for example, a paraffin and a naphthene).

5.3 Although a majority of the individual hydrocarbons present in petroleum distillates are determined, some co-elution of compounds is encountered. If this test method is utilized to determine bulk hydrocarbon group-type composition (PONA), the user of such data should be cautioned that some error will be encountered due to co-elution and a lack of identification of all components present. Samples containing significant amounts of olefinic or naphthenic, or both, constituents above octane may reflect significant errors in PONA-type groupings.

5.4 If water is or is suspected of being present, its concentration is determined by the use of Test Method **D1744**. Other compounds containing oxygen, sulfur, nitrogen, and so forth may also be present, and may co-elute with the hydrocarbons. When known co-elution exists, these are noted in the test method data tables. If determination of these specific compounds is required, it is recommended that test methods for

⁴ Johansen, N.G., and Ettre, L.S., "Retention Index Values of Hydrocarbons on Open Tubular Columns Coated with Methyl Silicone Liquid Phases," *Chromatographia*, Vol 5, No. 10, October 1982.

⁵ Johansen, N.G., Ettre, L.S., and Miller, R.L., "Quantitative Analysis of Hydrocarbons by Structural Group Type in Gasolines and Distillates. Part 1," *Journal of Chromatography*, Vol 256, 1983, pp. 393–417.

⁶ Kopp, V.R., Bones, C.J., Doerr, D.G., Ho, S.P., and Schubert, A.J., "Heavy Hydrocarbon/Volatility Study: Fuel Blending and Analysis for the Auto/Oil Air Quality Improvement Research Program," SAE Paper No. 930143, March 1993.

⁷ Schubert, A.J. and Johansen, N.J., "Cooperative Study to Evaluate a Standard Test Method for the Speciation of Gasolines by Capillary Gas Chromatography," SAE Paper No. 930144, March 1993.

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 The last approved version of this historical standard is referenced on www.astm.org.