



Designation: D2425 – 23

Standard Test Method for Hydrocarbon Types in Middle Distillates by Mass Spectrometry¹

This standard is issued under the fixed designation D2425; 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 an analytical scheme using the mass spectrometer to determine the hydrocarbon types present in conventional and synthesized hydrocarbons that have a boiling range of 160 °C to 343 °C (320 °F to 650 °F), 5 % to 95 % by volume as determined by Test Method D86. Samples with average carbon number value of paraffins between C₁₂ and C₁₆ and containing paraffins from C₁₀ and C₁₈ can be analyzed. Eleven hydrocarbon types are determined. These include: paraffins, noncondensed cycloparaffins, condensed dicycloparaffins, condensed tricycloparaffins, alkylbenzenes, indans or tetralins, or both, C_nH_{2n-10} (indenes, etc.), naphthalenes, C_nH_{2n-14} (acenaphthenes, etc.), C_nH_{2n-16} (acenaphthylenes, etc.), and tricyclic aromatics.

NOTE 1—This test method was developed on Consolidated Electro-dynamics Corporation Type 103 Mass Spectrometers. Operating parameters for users with a Quadrupole Mass Spectrometer are provided.

1.2 This test method is intended for use with full boiling range products that contain no significant olefin content.

Biodiesel (FAME components) could interfere with the separation of the sample and the characteristic mass fragments of FAME compounds are not defined in the procedure.

Hydrocarbons containing tertiary carbon fragments, sometimes found in synthetic aviation fuels, will interfere with the characteristic mass fragments of paraffins and result in a false, elevated cycloparaffin content.

NOTE 2—“No significant olefin content” for this method means <2.0 % by volume by Test Method D1319.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and deter-*

mine the applicability of regulatory limitations prior to use.

For a specific warning statement, see 11.1.

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

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

D1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption

D2549 Test Method for Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D6379 Test Method for Determination of Aromatic Hydrocarbon Types in Aviation Fuels and Petroleum Distillates—High Performance Liquid Chromatography Method with Refractive Index Detection

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions:*

3.1.1 This test method makes reference to many common gas chromatographic procedures and terms. Detailed definitions of these can be found in Practice E355 and Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

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 D02.04.0M on Mass Spectrometry.

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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.

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

3.2.1 *conventional hydrocarbons, n*—hydrocarbons derived from the following conventional sources: crude oil, natural gas liquid condensates, heavy oil, shale oil, and oil sands.

3.2.2 *synthesized hydrocarbons, n*—hydrocarbons derived from alternative sources such as coal, natural gas, biomass, and hydrogenated fats and oils by processes such as gasification, Fischer-Tropsch synthesis, and hydroprocessing.

4. Summary of Test Method

4.1 Samples are separated into saturate and aromatic fractions by liquid chromatography, and each fraction is analyzed by mass spectrometry. The analysis is based on the summation of characteristic mass fragments to determine the concentration of hydrocarbon types.

4.2 The summation of characteristic mass fragments are defined as follows:

$\sum 71$ (paraffins) = total peak height of m/e^+ 71 + 85.

$\sum 67$ (mono or noncondensed polycycloparaffins, or both) = total peak height of m/e^+ 67 + 68 + 69 + 81 + 82 + 83 + 96 + 97.

$\sum 123$ (condensed dicycloparaffins) = total peak height of m/e^+ 123 + 124 + 137 + 138 + ... etc. up to 249 + 250.

$\sum 149$ (condensed tricycloparaffins) = total peak height of m/e^+ 149 + 150 + 163 + 164 + ... etc. up to 247 + 248.

$\sum 91$ (alkyl benzenes) = total peak height of m/e^+ 91 + 92 + 105 + 106 + ... etc. up to 175 + 176.

$\sum 103$ (indans or tetralins, or both) = total peak height of m/e^+ 103 + 104 + 117 + 118 + ... etc. up to 187 + 188.

$\sum 115$ (indenes or C_nH_{2n-10} , or both) = total peak height of m/e^+ 115 + 116 + 129 + 130 + ... etc. up to 185 + 186.

128 (naphthalene) = total peak height of m/e^+ 128.

$\sum 141$ (naphthalenes) = total peak height of m/e^+ 141 + 142 + 155 + 156 + ... etc. up to 239 + 240.

$\sum 153$ (acenaphthenes or C_nH_{2n-14} , or both) = total peak height of m/e^+ 153 + 154 + 167 + 168 + ... etc. up to 251 + 252.

$\sum 151$ (acenaphthylenes or C_nH_{2n-16} , or both) = total peak height of m/e^+ 151 + 152 + 165 + 166 + ... etc. up to 249 + 250.

$\sum 177$ (tricyclic aromatics) = total peak height of m/e^+ 177 + 178 + 191 + 192 + ... etc. up to 247 + 248.

4.3 The average carbon numbers of the hydrocarbon types are estimated from spectral data. Calculations are made from calibration data dependent upon the average carbon number of the hydrocarbon types. The results of each fraction are mathematically combined according to their mass fractions as determined by the separation procedure. Results are expressed in mass percent.

5. Significance and Use

5.1 A knowledge of the hydrocarbon composition of process streams and petroleum products boiling within the range of 160 °C to 343 °C (320 °F to 650 °F) is useful in following the effect of changes in process variables, diagnosing the source of plant upsets, and in evaluating the effect of changes in composition on product performance properties.

5.2 A test method to determine total cycloparaffins and low level aromatic content is necessary to meet specifications for aviation turbine fuel containing synthesized hydrocarbons.

6. Interferences

6.1 Nonhydrocarbon types, such as sulfur and nitrogen-containing compounds, are not included in the matrices for this test method. If these nonhydrocarbon types are present to any large extent, (for example, mass percent sulfur >0.25) they will interfere with the spectral peaks used for the hydrocarbon-type calculation.

7. Sample Separation

7.1 Sample is to be separated into saturate and aromatic fractions. Liquid chromatography procedures based on Test Methods **D2549**, **D1319**, and **D6379** have been used.

NOTE 3—Test Method **D2549** is presently applicable only to samples having 5 % points of 232 °C (450 °F) or greater. Guidance on using Test Methods **D1319** and **D6379** is provided in the Annexes.

PROCEDURE A—MAGNETIC SECTOR SPECTROMETER

8. Apparatus

8.1 *Mass Spectrometer*—The suitability of the mass spectrometer to be used with this method of analysis shall be proven by performance tests described herein.

8.2 *Sample Inlet System*—Any inlet system permitting the introduction of the sample without loss, contamination, or change in composition. To fulfill these requirements it will be necessary to maintain the system at an elevated temperature in the range of 125 °C to 325 °C and to provide an appropriate sampling device.

8.3 *Microburet or Constant-Volume Pipet*.

9. Calibration

9.1 Calibration coefficients are attached which can be used directly provided:

9.1.1 Repeller settings are adjusted to maximize the m/e^+ 226 ion of *n*-hexadecane.

9.1.2 A magnetic field is used that will permit scanning from m/e^+ 40 to 292.

9.1.3 An ionization voltage of 70 eV and ionizing currents in the range 10 μ A to 70 μ A are used.

NOTE 4—The calibration coefficients were obtained for ion source