



Designation: D2425 – 19

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

1.3 *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.* For a specific warning statement, see 11.1.

1.4 *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.*

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 Spectroscopy.

Current edition approved Jan. 15, 2019. Published February 2019. Originally approved in 1965. Last previous edition approved in 2017 as D2425 – 17. DOI: 10.1520/D2425-19.

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

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products 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

3. Terminology

3.1 Definitions:

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

3.1.2 *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:

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

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

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

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

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

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

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

$\Sigma 115$ (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.

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

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

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

$\Sigma 177$ (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.

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 3—The calibration coefficients were obtained for ion source

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 2—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.

conditions such that the $\Sigma 67/\Sigma 71$ ratio for *n*-hexadecane was 0.26/1. The cooperative study of this test method indicated an acceptable range for this Σ ratio between 0.2/1 to 0.30/1.

10. Performance Test

10.1 Generally, mass spectrometers are in continuous operation and should require no additional preparation before analyzing samples. If the spectrometer has been turned on only recently, it will be necessary to check its operation in accordance with this method and instructions of the manufacturer to ensure stability before proceeding.

10.2 *Mass Spectral Background*—Samples in the carbon number range C_{10} to C_{18} should pump out so that less than 0.1 % of the two largest peaks remain. For example, background peaks from a saturate fraction at m/e^+ 69 and 71 should be reduced to less than 0.1 % of the corresponding peaks in the mixture spectrum after a normal pump out time of 2 min to 5 min.

11. Mass Spectrometric Procedure

11.1 *Obtaining the Mass Spectrum for Each Chromatographic Fraction*—Using a microburet or constant-volume

pipet, introduce sufficient sample through the inlet sample to give a pressure of 2 Pa to 4 Pa (15 mtorr to 30 mtorr) in the inlet reservoir. (**Warning**—Hydrocarbon samples of this boiling range are combustible.) Record the mass spectrum of the

sample from m/e^+ 40 to 292 using the instrument conditions outlined in 9.1.1 – 9.1.3.

PROCEDURE B—QUADRUPOLE SPECTROMETER

12. Apparatus

12.1 *Mass Spectrometer*—Mass spectrometers provided with a quadrupole as ion separator and use electron impact at 70 eV have been used.

12.2 *Sample Inlet System*—Any inlet system permitting the introduction of the sample without loss, contamination, or change in composition. Separation of components is not required.

12.2.1 For sample fractions that do not contain solvents:

12.2.1.1 The inlet to introduce the sample into the detector can be an all glass inlet system (also known as AGIS) connected to the GC inlet and interfaced with the mass spectrometer with uncoated tubing. The inlet system is installed in a gas chromatograph and heated at 300 °C, isothermal.

12.2.1.2 Gas chromatography using a boiling point column.

12.2.2 For sample fractions that contain solvents, gas chromatography using a boiling point column should be used to isolate the solvent.

12.2.3 Split ratio should be adjusted to prevent detector overload.

13. Calibration

13.1 Mass calibration is performed using PerFluoroTriButylAmine (PFTBA) which contains the masses 69, 131, 219, 414, and 502. An ionization voltage of 70 eV is used.

13.2 Tune the spectrometer using the tune file recommended by the manufacturer. Typically, the masses of 69, 219, 502 are used for tuning with 219 being the ion for repeller maximum. Ensure that none of the masses are saturated.

13.3 It is possible to check the tuning by introducing n-hexadecane and verifying that the sum of the $\sum 67/\sum 71 = 0.2 - 0.3$. Verify that the sum elements are those shown in 15.1, Eq 1 and Eq 2.

14. Mass Spectrometric Procedure

14.1 *Obtaining the Mass Spectrum for Each Chromatographic Fraction*—Using an automated sampling system or manual injection, inject an appropriate amount of each sample fraction. Record the mass spectrum of the sample from m/e^+ 50 to 300.

15. Calculations and Report

NOTE 4—A guideline for the calculations is available in Appendix X1.

15.1 *Aromatic Fraction*—Read peak heights from the record mass spectrum corresponding to m/e^+ ratios of 67 to 69, 71, 81 to 83, 85, 91, 92, 96, 97, 103 to 106, 115 to 120, 128 to 134, 141 to 148, 151 to 162, 165 to 198, 203 to 212, 217 to 226, 231 to 240, 245, 246, 247 to 252.

Find:

TABLE 1 Parent Ion Isotope Factors and Mole Sensitivities

Carbon No.	m/e	Isotope Factor, K_1	Mole Sensitivity, K_2
Alkylbenzenes			
10	134	0.1101	85
11	148	0.1212	63
12	162	0.1323	60
13	176	0.1434	57
14	190	0.1545	54
15	204	0.1656	51
16	218	0.1767	48
17	232	0.1878	45
18	246	0.1989	42
		L_1	L_2
Naphthalenes			
11	142	0.1201	194
12	156	0.1314	166
13	170	0.1425	150
14	184	0.1536	150
15	198	0.1647	150
16	212	0.1758	150
17	226	0.1871	150
18	240	0.1982	150

$$\sum 71 = 71 + 85 \quad (1)$$

$$\sum 67 = 67 + 68 + 69 + 81 + 82 + 83 + 96 + 97 \quad (2)$$

$$\sum 91 = \sum_{N=0}^{N=6} [(91 + 14N) + (92 + 14N)] \quad (3)$$

$$\sum 103 = \sum_{N=0}^{N=6} [(103 + 14N) + (104 + 14N)] \quad (4)$$

$$\sum 115 = \sum_{N=0}^{N=5} [(115 + 14N) + (116 + 14N)] \quad (5)$$

$$\sum 141 = \sum_{N=0}^{N=7} [(141 + 14N) + (142 + 14N)] \quad (6)$$

$$\sum 153 = \sum_{N=0}^{N=7} [(153 + 14N) + (154 + 14N)] \quad (7)$$

$$\sum 151 = \sum_{N=0}^{N=7} [(151 + 14N) + (152 + 14N)] \quad (8)$$

$$\sum 177 = \sum_{N=0}^{N=5} [(177 + 14N) + (178 + 14N)] \quad (9)$$

15.2 Calculate the mole fraction at each carbon number of the alkylbenzenes for $n = 10$ to $n = 18$ as follows:

$$\mu_n = [P_m - P_{m-1}(K_1)]/K_2 \quad (10)$$

where:

μ_n = mole fraction of each alkylbenzene as represented by n which indicates the number of carbons in each molecular species.

m = molecular weight of the alkylbenzene being calculated,

$m - 1$ = molecular weight minus 1,

P = polyisotopic mixture peak at $m, m - 1$,

K_1 = isotopic correction factor (see Table 1), and

K_2 = mole sensitivity for n (see Table 1).

NOTE 5—This step of calculation assumes no mass spectral pattern contributions from other hydrocarbon types to the parent and parent-1 peaks of the alkylbenzenes. Selection of the lowest carbon number 10 is

based upon the fact that C₉ alkylbenzenes boil below 204°C (400°F) and their concentration can be considered negligible.

15.3 Find the average carbon number of the alkylbenzenes, A, in the aromatic fraction as follows:

$$A = \left(\sum_{n=10}^{n=18} n \times \mu_n \right) / \left(\sum_{n=10}^{n=18} \mu_n \right) \quad (11)$$

15.4 Calculate the mole fraction at each carbon number of the naphthalenes for $n = 11$ to $n = 18$ as follows:

$$x_n = [P_m - P_{m-1}(L_1)]/L_2 \quad (12)$$

where:

x_n = mole fraction of each naphthalene as represented by n which indicates the number of carbons in each molecular species,

m = molecular weight of the naphthalenes being calculated,

$m - 1$ = molecular weight minus 1,

P = polyisotopic mixture peak at m , $m - 1$,

L_1 = isotopic correction factor (see Table 1), and

L_2 = mole sensitivity for n (see Table 1).

NOTE 6—This step of calculation assumes no mass spectral pattern contributions to the parent and parent-1 peaks of the naphthalenes. The concentration of naphthalene itself at a molecular weight of 128 shall be determined separately from the polyisotopic peak at $m/e^+ 128$ in the matrix calculation. The average carbon number for the naphthalenes shall be calculated from carbon number 11 (molecular weight 142) to 18 (molecular weight 240).

15.5 Find the average carbon number of the naphthalenes, B, in the aromatic fraction as follows:

$$B = \left(\sum_{n=11}^{n=18} n x_n \right) / \left(\sum_{n=11}^{n=18} x_n \right) \quad (13)$$

15.6 Selection of pattern and sensitivity data for matrix carbon number of the types present. The average carbon number of the paraffins and cycloparaffins ($\Sigma 71$ and $\Sigma 67$, respectively) are related to the calculated average carbon of the alkylbenzenes (15.3), as shown in Table 2. Both $\Sigma 71$ and $\Sigma 67$ are included in the aromatic fraction matrix to check on possible overlap in the separation. The other types present, represented by Σ 's 103, 115, 153, and 151, are usually relatively low in concentration so that their parent ions are affected by other types present. The calculation of their average carbon number is not straight forward. Therefore, their average carbon numbers are estimated by inspection of the aromatic spectrum. Generally, their average carbon numbers may be taken to be equivalent to that of the naphthalenes, or to the closest whole number thereof, as calculated in 15.5. The average carbon number of tricyclic aromatics $\Sigma 177$ has to be at least C₁₄ and in full boiling range middle distillates C₁₄ may be used to represent the $\Sigma 177$ types carbon number. From the calculated and estimated average carbon numbers of the

hydrocarbon types, a matrix for the aromatic fraction is set up using the calibration data given in Table 3. A sample matrix for the aromatic fraction is shown in Table 4. The matrix calculations consist in solving a set of simultaneous linear equations. The pattern coefficients are listed in Table 3. The constants are the Σ values determined from the mass spectrum. Second approximation solutions are of sufficient accuracy. If many analyses are performed using the same type of a matrix, the matrix may be inverted for simpler, more rapid desk calculation. Matrices may also be programmed for automatic computer operations. The results of matrix calculations are converted to mass fractions by dividing by mass sensitivity. The mass fractions are normalized to the mass percent of the aromatic fraction, as determined by the separation procedure.

15.7 *Saturate Fraction*—Read peak at heights from the record of the mass spectrum corresponding to m/e^+ ratios of 67 to 69, 71, 81 to 83, 85, 91, 92, 96, 97, 105, 106, 119, 120, 123, 124, 133, 134, 137, 138, 147 to 152, 161 to 166, 175 to 180, 191 to 194, 205 to 208, 219 to 222, 233 to 236, 247 to 250.

Find:

$$\Sigma 71 = 71 + 85 \quad (14)$$

$$\Sigma 67 = 67 + 68 + 69 + 81 + 82 + 83 + 96 + 97 \quad (15)$$

$$\Sigma 123 = \sum_{N=0}^{N=9} [(123 + 14N) + (124 + 14N)] \quad (16)$$

$$\Sigma 149 = \sum_{N=0}^{N=7} [(149 + 14N) + (150 + 14N)] \quad (17)$$

$$\Sigma 91 = \sum_{N=0}^{N=6} [(91 + 14N) + (92 + 14N)] \quad (18)$$

15.8 Selection of the pattern and sensitivity data for matrix calculation is dependent upon the average carbon number of the types present. The average carbon number of the paraffins and cycloparaffin types (Σ 's 71, 69, 123, and 149), are related to the calculated average carbon number of the alkylbenzenes of the aromatic fraction (15.3), as shown in Table 2. The $\Sigma 91$ is included in the saturate fraction as a check on the efficiency of the separation procedure. The pattern and sensitivity data for the $\Sigma 91$ are based on the calculated or estimated average carbon number from the mass spectra of the aromatic fraction (see 15.3). From the determined average carbon numbers of the hydrocarbon types, a matrix for the saturate fraction is set up using the calibration data given in Table 3. A sample matrix for the saturate fraction is shown in Table 5. The matrix calculations of the saturate fraction consists in solving a set of simultaneous linear equations. The results of the matrix calculations (second approximation solutions are sufficient) are converted to mass fractions by dividing by mass sensitivity. The mass fractions are normalized to the mass percent of the saturate fraction as determined by the separation procedure.

15.9 Report the mass percent of each hydrocarbon type to the nearest 0.1 % and reference this test method and procedure used.

TABLE 2 Relationship Between Average Carbon Numbers of Alkylbenzenes, Paraffins, and Cycloparaffins

Alkylbenzenes	Paraffin and Cycloparaffin
Average Carbon No.	Average Carbon No.
10	11
11	12
12	13
13	15 (14.5)
14	16 (15.5)

16. Precision and Bias

16.1 *Procedure A*—The precision of this test method as obtained by statistical examination of interlaboratory test results on samples having the composition given in Table 6 is as follows:

TABLE 3 Patterns and Sensitivities for Middle Distillates

Hydrocarbon Type	Paraffins				Noncondensed Cycloparaffins				Condensed Dicycloparaffins			Condensed Tricycloparaffins		
Carbon No...	12	13	14.5	15.5	12	13	14.5	15.5	13	14.5	15.5	13	14.5	15.5
Peaks read:														
∑71	100	100	100	100	4	4	6	6	2	1.1	1.5	1	1	2
∑67	19	21	23	26	100	100	100	100	160	130	150	175	170	150
∑123	0.1	0.2	1	1	1	3	100	100	100	26	10	20
∑149	0.2	5	8	100	100	100
∑91 to 176	0.4	0.4	0.4	0.4	0.2	3	4	4	5	15	15	20
∑103 to 188	1	...	3
∑115 to 186	0.5	1	1	1	1	0.5
∑128 pk
∑141	9	9	10	12	2	0.3	0.2	0.1	0.1	0.4
∑153	1	2	2	2
∑151	1	5	7	10
∑177	2	2
Sensitivity:														
Mole	148	170	192	238	302	347	416	439	220	268	298	220	268	298
Volume	66	70	74	81	145	153	165	170	107	137	117	118	150	127
Mass	87	92	97	104	180	191	204	209	122	156	134	124	158	135
Hydrocarbon Type	Alkylbenzenes				Indans or Tetralins, or Both				Indenes or C _n H _{2n-10} , or Both			Naphthalenes		
Carbon No...	11	12	13	14	10	11	12	13	10	13	10	11	12	13
Peaks read:														
∑71	0.3	0.3	0.4	0.5	0.2	0.4	0.4	1	0.3	1.7	0.5	5.2	1.5	2
∑67	0.7	0.7	2	3	0.6	1	1	2	0.3	6.0	0.8	1.2	1.5	2
∑123	0.1	0.1	0.2	0.3	...	0.1	1	2	0.4	4.8	0.2	0.5	7.8	4
∑149	1.3	1	1.5	2	...	0.1	0.2	0.3	...	0.9	...	0.1	0.7	0.5
∑91 to 176	100	100	100	100	15 to 34 ^{A,B}	18	17	15	0.6	6.2	0.1	0.9	1	1
∑103 to 188	9	10	10	9	100	100	100	100	1.5	20.3	0.6	0.1	0.1	0.1
∑115 to 186	4.4	4.5	5	5	20 to 12 ^{A,B}	28	25	25	100	100	11.4	23	19	18
∑128 pk	0.7	1	1	1	3	5.4	7	...	15	13	100	0.7	5.6	5.6
∑141	1.0	2.5	28	...	100	100	100
∑153	6.1	8	10
∑151	4.5	7	7
∑177	0.6
Sensitivity:														
Mole	450	450	450	450	380	420	420	420	410	372	236	360	380	380
Volume	265	242	222	206	280	276	250	227	307	198	211	259	248	226
Mass	304	278	256	237	288	288	263	241	315	200	184	254	244	224
Hydrocarbon Type	Acenaphthenes or C _n H _{2n-14} , or Both		Acenaphthylenes or C _n H _{2n-16}		Tricyclic Aromatics	Characteristic Mass Groupings								
Carbon No...	12	13	12	13	14	Peaks Read				Hydrocarbon Types				
Peaks read:														
∑71	1	1	1	1	0.6									
∑67	0.3	2	1	5	0.7	∑71 = 71, 85				paraffins				
∑91 to 176	0.1	5	1	3	18	∑67 = 67, 68, 69, 81, 82, 83, 96, 97				cycloparaffins, mono or noncondensed				
∑103 to 188	...	3	0.2	3	1.5					cycloparaffins				
∑115 to 186	0.8	0.8	0.3	2.7	1.0	∑123 = 123, 134, 137, 138 up to 249, 250				condensed dicycloparaffins				
∑128 pk	1	0.7	0.2	0.1	0.8	∑149 = 149, 150, 163, 164 up to 247, 248				condensed tricycloparaffins				
∑141	8	10	1	...	0.3	∑91 = 91, 92, 105, 106 up to 175, 176				alkylbenzenes				
∑153	100	100	17	15	3.5	∑103 = 103, 104, 117, 118, up to 187, 188				indan or tetralins, or both				
∑151	27	20	100	100	30	∑115 = 115, 116, 129, 130 up to 185, 186				C _n H _{2n-10} (indenes, etc.)				
∑177	...	4	...	15	100	∑128 = poly 128 pk				naphthalene				
Sensitivity:														
Mole	330	330	340	340	365	∑141 = 141, 142, 155, 156 up to 239, 240				naphthalenes				
Volume	218	198	199	187	211	∑153 = 153, 154, 167, 168 up to 251, 252				C _n H _{2n-14} (acenaphthenes, etc.)				
Mass	214	196	224	205	205	∑151 = 151, 152, 165, 166 up to 249, 250				C _n H _{2n-16} (acenaphthylenes, etc.)				
						∑177 = 177, 178, 191, 192 up to 247, 248				tricyclic aromatics				

^A = methyl indans.

^B tetralins.

16.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test

material, would be in the long run, in the normal and correct operation of the test method, exceed the values shown in Table 7 only in one case in twenty.

TABLE 4 Aromatic Concentration Matrix

Hydrocarbon Type	Paraffins	Cycloparaffins	Alkylbenzenes	Indans and Tetralins	Indenes	Naphthalene	Naphthalenes	Acenaphthenes C _n H _{2n-14}	Acenaphthylenes C _n H _{2n-16}	Tricyclic Aromatics
Carbon No.	15.5	15.5	14	13	13	10	13	13	13	14
Peaks read:										
Σ71	100	6	0.5	1	1.7	0.5	2	1	1	0.6
Σ67	26	100	3	2	6	0.8	2	2	5	0.7
Σ91	0.4	3	100	15	6.2	0.1	1	5	3	18
Σ103	...	2	9	100	20.3	0.6	0.1	3	3	1.5
Σ115	...	1	5	25	100	11.4	18	0.8	2.7	1
Σ128 pk	1	3	13	100	5.6	0.7	0.1	0.8
Σ141	12	0.3	28	...	100	10	...	0.3
Σ153	...	2	6.1	...	10	100	15	3.5
Σ151	...	10	4.5	...	7	20	100	30
Σ177	...	2	0.6	4	15	100
Sensitivity:										
Mole	238	439	450	420	372	236	380	330	340	365
Volume	81	170	206	227	198	211	226	198	187	211
Weight	105	209	237	241	200	184	224	196	205	205

TABLE 5 Saturate Concentration Matrix

Hydrocarbon Type	Paraffins	Monocyclo-paraffins	Dicyclo-paraffins	Tricyclo-paraffins	Alkyl-benzenes
Carbon No.	15.5	15.5	15.5	15.5	14
Σ71	100	6	1.5	2	0.5
Σ67	26	100	150	150	3
Σ123	0.2	3	100	20	0.3
Σ149	8	100	2
Σ91	0.4	3	5	20	100
Sensitivity:					
Mole	238	439	298	298	450
Volume	81	170	117	127	206
Weight	105	209	134	135	237

TABLE 6 Composition of Samples Tested^A

Component	Mean, Mass, %	σ_r^B	σ_R^C
Sample No. 7 ^D :			
Paraffins	44.25	0.16	1.30
Monocycloparaffin	22.04	0.34	1.70
Dicycloparaffin	8.54	0.23	1.42
Tricycloparaffin	2.84	0.11	0.64
Alkylbenzene	0.33	0.04	0.10
Sample No. 8 ^E :			
Paraffins	0.07	0.14	0.14
Cycloparaffin	0.75	0.15	0.25
Alkylbenzene	5.10	0.10	0.44
Indan and/or tetralin	3.65	0.09	0.14
C _n H _{2n-10}	2.05	0.08	0.20
Naphthalenes	5.15	0.08	0.29
C _n H _{2n-14}	2.50	0.04	0.28
C _n H _{2n-16}	1.65	0.10	0.18
C _n H _{2n-18}	1.05	0.04	0.14

TABLE 7 Precision of Test Method

Compound	Concentration Mass, %	Repeatability	Reproducibility
Saturate Fraction:			
Paraffins	40 to 50	0.5	4.0
Monocycloparaffins	18 to 25	1.1	5.2
Dicycloparaffins	6 to 12	0.7	4.4
Tricycloparaffins	1 to 5	0.3	2.0
Alkylbenzenes	0 to 3	0.2	0.3
Aromatic Fraction:			
Paraffins	0 to 2	0.4	0.6
Cycloparaffins	0 to 2	0.5	0.9
Alkylbenzenes	3 to 8	0.3	1.4
Indan and/or tetralins	2 to 5	0.3	0.5
C _n H _{2n-10}	0 to 4	0.3	0.7
Naphthalenes	3 to 8	0.3	1.0
C _n H _{2n-14}	0 to 3	0.1	0.9
C _n H _{2n-16}	0 to 3	0.3	0.7
C _n H _{2n-18}	0 to 3	0.1	0.4

^A Twelve laboratories cooperated and each sample was run twice.
^B σ_r = repeatability standard deviation.
^C σ_R = reproducibility standard deviation.
^D Sample No. 7 = saturate fraction of a virgin middle distillate (78.0 wt % of total).
^E Sample No. 8 = aromatic fraction of a virgin middle distillate (22.0 wt % of total).

NOTE 8—The precision for this test method was not obtained in accordance with RR:D02-1007.

16.2 Procedure B:

16.2.1 The precision of this test method is to be determined by an interlaboratory study (ILS).

16.2.2 An interim repeatability study was conducted following the guidelines given in Practice D6300, section 6.2.1. The sample set included diesel fuels, European gas oils, jet fuels, and renewable fuels. A proper ILS will be completed by 2022.

16.2.3 Reproducibility—Reproducibility has not been determined.

16.1.2 Reproducibility—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values shown in Table 7 only in one case in twenty.

NOTE 7—If samples are analyzed that differ appreciably in composition from those used for the interlaboratory study, this precision statement may not apply.