



Designation: D8071 – 17

# Standard Test Method for Determination of Hydrocarbon Group Types and Select Hydrocarbon and Oxygenate Compounds in Automotive Spark-Ignition Engine Fuel Using Gas Chromatography with Vacuum Ultraviolet Absorption Spectroscopy Detection (GC-VUV)<sup>1</sup>

This standard is issued under the fixed designation D8071; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This test method is a standard procedure for the determination of paraffins, isoparaffins, olefins, naphthenes, aromatics, ethanol, and methanol in automotive spark-ignition engine fuels using gas chromatography and vacuum ultraviolet detection (GC-VUV).

1.2 Concentrations of compound classes and certain individual compounds are determined by percent mass or percent volume.

1.2.1 The method is developed for testing automotive spark-ignition engine fuels having concentrations of approximately 6 % to 17 % by mass paraffins, 24 % to 70 % by mass isoparaffins, 0.1 % to 16 % by mass olefins, 1 % to 14 % by mass naphthenes, and 16 % to 58 % by mass aromatics. Pending confirmation by a full interlaboratory study, the method is projected to be applicable to a wider range of concentrations and matrices. Temporary repeatability has been determined on a limited subset of samples given in 17.1.

1.3 Individual aromatic components and groups of aromatics greater than 0.01 % by mass are determined.

1.3.1 The method is developed for testing automotive spark-ignition engine fuels having concentrations of 0.08 % to 2.2 % by mass benzene, 1.7 % to 15 % by mass toluene, 0.3 % to 3 % by mass ethylbenzene, 1.5 % to 17 % by mass total xylenes, 0.06 % to 0.6 % by mass naphthalene, and 0.06 % to 1.1 % by mass total methyl-naphthalenes. Pending confirmation by a full interlaboratory study, the method is projected to be applicable to a wider range of concentrations and matrices. Temporary

repeatability has been determined on a limited subset of samples given in Table 8.

1.4 *Isooctane* (2,2,4-trimethylpentane) content is determined in the range 0.1 % by mass to 22 % by mass.

1.5 Ethanol and methanol may be determined by this test method. Ethanol is determined in the range of 0.1 % by mass to 15.0 % by mass. Methanol is determined in the range of 0.1 % by mass to 1.0 % by mass. Temporary repeatability has been determined on a limited subset of samples containing ethanol given in 17.1. Repeatability for methanol is currently not available.

1.6 Individual hydrocarbon components are typically not baseline-separated by the procedure described in this test method, that is, some components will coelute. The coelutions are resolved at the detector using VUV absorbance spectra and deconvolution algorithms.

1.7 While this test method reports percent mass and percent volume for several specific components that may be present in automotive spark-ignition engine fuel, it does not attempt to speciate all possible components that may occur in automotive spark-ignition engine fuel. In particular, this test method is not intended as a type of detailed hydrocarbon analysis (DHA).

1.8 This test method has been tested for spark-ignition engine fuels; the method may apply to spark-ignition blending streams but has not been extensively tested for such applications.

1.9 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

<sup>1</sup> 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 D02.04.0L on Gas Chromatography Methods.

Current edition approved March 1, 2017. Published March 2017. DOI: 10.1520/D8071-17.

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*:<sup>2</sup>

**D4057** Practice for Manual Sampling of Petroleum and Petroleum Products

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

**D5842** Practice for Sampling and Handling of Fuels for Volatility Measurement

**D6299** Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

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

**D6730** Test Method for Determination of Individual Components in Spark Ignition Engine Fuels by 100–Metre Capillary (with Precolumn) High-Resolution Gas Chromatography

**D6792** Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard*:

3.1.1 *integration filter, n*—a mathematical operation performed on an absorbance spectrum for the purpose of converting the spectrum to a single-valued response suitable for representation in a two-dimensional chromatogram plot.

3.1.2 *library reference spectrum, n*—an absorbance spectrum representation of a molecular species stored in a library database and used for identification of a compound/compound class or deconvolution of multiple coeluting compounds.

3.1.3 *response area, n*—generally refers to a response summed over a given time interval and has units of absorbance units (AU).

3.1.3.1 *Discussion*—A time factor necessary to convert a response area to a true mathematical area cancels out of all critical calculations and is omitted.

3.2 *Abbreviations*:

3.2.1 *AU*—absorbance units

3.2.2 *DHA*—detailed hydrocarbon analysis

3.2.3 *GC-VUV*—gas chromatography with vacuum ultraviolet spectroscopy detection

3.2.4 *RI*—retention index

3.2.5 *RRF*—relative response factor

## 4. Summary of Test Method

4.1 An automotive spark-ignition fuel sample is introduced to a gas chromatographic (GC) system. After volatilization, the

effluent is introduced onto a GC column for separation, and then detected by a vacuum ultraviolet absorption spectroscopy detector.<sup>3</sup> The separation is accomplished using a 30 m, non-polar phase capillary column and a moderately fast temperature ramp (typical operating parameters of this test method are given in Section 14). Coelutions are resolved by the detector using vacuum ultraviolet absorbance spectra and deconvolution.

4.2 The result of the measurement is the determination of the total response areas of the five hydrocarbon classes of paraffins, isoparaffins, olefins, naphthenes, and aromatics, in addition to several individual species components. The percent mass concentrations are calculated from the response areas using class-based or compound-specific relative response factors, as appropriate.

## 5. Significance and Use

5.1 The determination of class group composition of automotive spark-ignition fuels as well as quantification of various individual species such as oxygenates and aromatics in automotive fuels is useful for evaluating quality and expected performance, as well as compliance with various governmental regulations.

## 6. Interferences

6.1 Interferences with this test method, if any, have not been determined.

## 7. Apparatus

7.1 *Gas chromatograph*, equipped with automated oven temperature control and split/splitless inlet.

7.1.1 Preferably the gas chromatograph is equipped with an autosampler. This is not strictly necessary, but all statistical data were obtained using a GC equipped with an autosampler.

7.1.2 *Syringe*, 0.5 uL volume appropriate for injecting liquid samples.

7.2 *Carrier Gas*, for gas chromatograph: nitrogen, helium, or hydrogen (see 8.2).

7.3 *Purge/Makeup Gas*, for detector: nitrogen or argon (see 8.3).

7.4 *Oxygen, Water, Hydrocarbon Filters*, to further purify GC carrier gas and detector purge/makeup gas.

7.5 *Capillary Analytical Column*, nonpolar (for example, dimethyl polysiloxane) phase, dimensions 30 m length, 0.25 mm internal diameter, 0.25 μm film thickness.

7.6 *Vacuum Ultraviolet Absorption Spectroscopy Detector*, capable of measuring 125 nm to 240 nm absorbance spectra with a wavelength resolution of 1 nm or better.

7.6.1 The detector shall be able to interface with a gas chromatographic system and measure an eluent with a scan frequency of at least 5 Hz with a baseline peak-to-peak noise

<sup>2</sup> 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.

<sup>3</sup> The sole source of supply of the apparatus known to the committee at this time is VUV-Analytics, Cedar Park, Texas. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

width over a 10 s interval no greater than 0.002 AU when averaged over the following wavelength regions: 125 nm to 240 nm, 170 nm to 200 nm, 125 nm to 160 nm, and 0.001 AU when averaged over the 140 nm to 160 nm wavelength region.

7.6.2 The detector shall be equipped with a shutter or equivalent mechanism that allows the detector array to be blocked from the light source in order to perform a “dark” measurement of electronic noise level.

7.6.3 The detector shall be equipped with a flow cell capable of being heated to at least 275 °C.

7.6.4 The detector shall have an independently controlled makeup gas capability, capable of providing up to 5 mL/min additional flow of nitrogen, helium, or argon to the flow cell.

7.7 *Data Processing System*, capable of storing and processing absorbance scan data and corresponding time.

7.7.1 Data processing system shall include a database library of vacuum ultraviolet reference spectra, compound class information, carbon number, density, and approximate retention index values. Data processing system shall also store relative response factors for each hydrocarbon class in addition to relative response factors for individually reported compounds.

7.7.2 Data processing system shall be capable of implementing equations and fit procedures that result in deconvolution of absorbance spectra that contain contributions from multiple species.

7.7.3 Data processing system shall be capable of binning and storing response contributions from each deconvolution analysis and reporting a combined total response at the end of the analysis.

7.7.4 Data processing system shall be capable of implementing equations to convert response areas to percent mass and further convert percent mass to percent volume.

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Nitrogen, helium, or hydrogen carrier gas for gas chromatograph, 99.999 % pure.

8.3 Nitrogen, helium, or argon purge/makeup gas for vacuum ultraviolet detector, 99.999 % pure.

8.4 Methylene chloride, reagent grade, used as a solvent test sample and GC rinse solvent. (**Warning**—Toxic material. May be combustible at high temperatures.)

8.5 Response test sample, 500 ug/mL benzene in methanol solvent.

8.6 Sample consisting of C<sub>4</sub> to C<sub>15</sub> linear alkanes, used as retention time markers (optional).

8.7 A system validation mixture that complies with Practice **D4307**, having the components and approximate concentrations given in **Table 1**. The concentrations of the prepared

**TABLE 1 System Validation Mixture**

Component	Concentration (percent mass)
Cyclopentane	1.1
n-Pentane	1.1
Cyclohexane	2.1
2,3-Dimethylbutane	2.1
n-Hexane	2.1
1-Hexene	1.5
Methylcyclohexane	4
4-Methyl-1-hexene	1.6
n-Heptane	3.5
1,2-Dimethylcyclohexane	5
Isooctane	5
n-Octane	5
1,2,4-Trimethylcyclohexane	4
n-Nonane	4.5
n-Decane	4.5
n-Undecane	3.5
n-Dodecane	3.5
Benzene	2.2
Toluene	2.2
trans-Decahydronaphthalene	4
n-Tetradecane	4.5
Ethylbenzene	4.5
o-Xylene	4
n-Propylbenzene	5
1,2,4-Trimethylbenzene	4.5
1,2,3-Trimethylbenzene	5
1,2,4,5-Tetramethylbenzene	5
Pentamethylbenzene	5
Total Paraffins	32.2
Total Isoparaffins	7.1
Total Olefins	3.1
Total Naphthenes	20.2
Total Aromatics	37.4
Total Xylenes	4.0

system validation mixture should be close to those in **Table 1** and shall otherwise be accurately known.

8.7.1 The components of the system validation mixture may be modified to include other components of particular relevance to the method. For example, the system validation mixture may include ethanol or methylnaphthalene, or both.

8.7.2 The components of the system validation mixture may be modified to include linear alkanes or other compounds necessary for determining a retention time marker list (see **12.1** and **12.2**).

8.7.3 The concentrations in **Table 1** may be suitably modified to accommodate additional or substituted components. The concentrations shall otherwise be accurately known.

8.8 A quality control (QC) sample, consisting of automotive spark-ignition engine fuel, similar in characteristics to samples that are to be routinely analyzed. The QC sample should be routinely analyzed and the results compared to their values obtained on initial setup of the instrumentation or replenishment of the QC stock supply. See **Note 1**.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

NOTE 1—Refer to Practices [D6299](#) and [D6792](#) for guidance on quality assurance (QA) practices.

## 9. Hazards

9.1 Many of the compounds in automotive spark-ignition engine fuel or other test samples used in this test method are toxic, flammable, or both. Safety and sample-handling procedures appropriate for working with such materials shall be in place before attempting to use this test method.

9.2 Hydrogen is flammable and potentially explosive if not properly used. Use of hydrogen as a GC carrier gas shall only be done at laboratories experienced with its use, with proper safety procedures in place.

## 10. Sampling

10.1 Refer to Practices [D4057](#) and [D5842](#) for guidelines on obtaining automotive spark-ignition engine fuel samples for analysis. Samples should be kept refrigerated at approximately 4 °C until being measured. A small amount (between ~0.1 mL and 1 mL) of sample should be drawn from the refrigerated stock sample and placed in a pre-cooled, sealed autosampler vial. The autosampler vial should be refrigerated at approximately 4 °C until just prior to measurement.

## 11. Preparation of Apparatus

11.1 Ensure that all gas connections are properly made, without leaks.

11.2 Install oxygen, moisture, and hydrocarbon filters in gas lines upstream of GC and detector. Maintain gas filters as instructed by manufacturer.

11.3 Install the 30 m column in the GC inlet. Condition the column according to the column manufacturer's recommendations prior to installation in the detector.

11.4 Perform maintenance on the GC as suggested by manufacturer, such as replacing septum and liner.

11.5 Configure the injector, carrier gas, and other GC parameters according to [Table 2](#).

11.6 Inject the response test sample defined in [8.5](#) according to the conditions in [Table 2](#). Verify that the total (summed)

response of the benzene peak, where the response consists of average 170 nm to 200 nm absorbance, is between 0.8 AU and 1.2 AU.

## 12. Calibration and Standardization

12.1 On installation of GC-VUV apparatus, after significant maintenance of GC-VUV apparatus, or after a significant method change, run the linear alkane sample using the same flow conditions and oven ramp profile as measured samples (optional—see [12.2.1](#); see [Section 14](#) for recommended run conditions). Note retention times of C<sub>4</sub> to C<sub>15</sub> linear alkanes. These will serve as retention time markers.

12.1.1 Significant method changes include changing the GC, column type, carrier gas type, or oven ramp profile. Significant maintenance of the GC-VUV apparatus includes changing or trimming the analytical column.

12.2 A list of retention times and retention indices for the linear alkanes is used to estimate rough elution times of other compounds in the VUV library according to an interpolation scheme. The most convenient retention index scheme sets the linear alkane retention indices to multiples of 100 according to carbon number: butane RI = 400, pentane RI = 500, etc. Each compound entry in the VUV library shall have an associated retention index generated using the same RI scheme. Otherwise, the associated retention indices do not need to be particularly accurate. The RI values for nonpolar capillary chromatography found in the literature or other ASTM test methods, such as Test Method [D6730](#), may be used.

12.2.1 The retention time markers do not have to be linear alkanes. A retention time marker list of convenient compounds, retention times, and approximate retention indices meeting the following conditions may be used:

12.2.1.1 The markers shall approximately span the total analysis time and have RI spacing of approximately 100 or less.

12.2.1.2 The markers may be chosen to be compounds whose VUV spectra are particularly distinct and easy to pick out of a GC-VUV automotive spark-ignition engine fuel data set.

12.2.2 In lieu of a C<sub>4</sub> to C<sub>15</sub> linear alkane standard, a C<sub>5</sub> to C<sub>15</sub> linear alkane standard may be used.

12.2.2.1 Butane may be added to the retention marker list using another standard or automotive spark-ignition fuel sample that contains butane. Butane elutes early in the chromatogram and can be identified by its absorbance spectrum.

12.2.3 If water is present, it may be added to the retention marker list with RI of 323. If propane is present, it may be added to the retention marker list with an RI of 300. Propane and water, if present, can be found at the beginning of a gasoline chromatogram and partially co-elute.

12.2.4 Iso-butane may be included with a RI of 360. It elutes between propane/water and butane.

12.2.5 Ethanol may be added with a RI of 441.

12.2.6 Methanol may be added with a RI of 375.

12.2.7 The RI values given for water, methanol, ethanol, and iso-butane are approximate, and may vary with varying GC conditions and concentrations. More precise RI values may

**TABLE 2 Instrument Settings for Response Test Sample**

Column type, dimensions	Capillary, 30 m × 0.25 mm ID × 0.25 μm film thickness
Column phase	Nonpolar (for example, 100 % dimethyl polysiloxane)
Injector temperature	250 °C
Injection volume	0.3 μL
Split ratio	20:1
Column flow	1 mL/min
Oven initial temperature/wait	35 °C/2 min
Oven ramp	15 °C/min
Final oven temperature	200 °C
Final hold time	0 min
Detector makeup gas pressure (gauge)	1.72 kPa
Detector scan time	11 ms
Detector averages per scan	20
Detector flow cell temperature	275 °C
Transfer tube temperature	275 °C

be obtained for a given configuration by analyzing appropriate standards. A linear interpolation scheme is sufficient.

12.2.8 Once updated, the same retention time marker list is used for all subsequent automotive spark-ignition fuel measurements until the next modification or maintenance of the GC-VUV instrumentation.

12.3 The conversion from response areas to percent mass uses class-based or compound-specific relative response factors. The relative response factors account for the differing areal response per unit mass for the various hydrocarbon classes.

12.4 For the purpose of this calculation, the response at a given elution time refers to the absorbance averaged over the 125 nm to 240 nm wavelength region. The response area refers to the sum of the response over all detector scans within a given time region. A true area can be generated by multiplying this quantity by the time interval between scans. However, this step is unnecessary when the scan rate is kept constant throughout a given measurement. For the purposes of this test method, the response area is taken to be a sum having units of absorbance units.

12.5 The response factors are relative to the response of methane, which is taken to have a relative response factor of 1.

12.6 Relative response factors used to obtain the statistical data in this test method are given in [Table 3](#) and [Table 4](#), and are suitable for use with this test method.

12.7 Relative response factors may alternatively be refined or determined by one of the following procedures:

12.7.1 The relative response factor for a given compound may be determined from the compound's absorption cross section (expressed in  $\text{cm}^2/\text{molecule}$ ) and methane's cross section by calculating:

$$RRF_a = \frac{\Sigma_{\text{methane}}}{MW_{\text{methane}}} \times \frac{MW_a}{\Sigma_a} \quad (1)$$

where:

$RRF_a$  = relative response factor for compound  $a$ ,  
 $MW$  = molecular weight, and  
 $\Sigma$  = absorption cross section averaged over the 125 nm to 240 nm wavelength region.

12.7.2 The relative response factor for a given compound may be determined from a prepared standard consisting of the compound and a second compound whose relative response factor is known. The relative amounts of the two compounds in the standard must be known:

$$RRF_2 = \frac{M_2 \cdot A_1}{M_1 \cdot A_2} RRF_1 \quad (2)$$

**TABLE 3 Relative Response Factors for Bulk Hydrocarbon Classes**

Hydrocarbon Class	Relative Response Factor
Paraffin	0.769
Isoparaffin	0.781
Olefin	0.465
Naphthene	0.786
C <sub>9</sub> + Aromatics	0.296

**TABLE 4 Relative Response Factors for Specific Individual Compounds and Compound Groups**

Compound	Relative Response Factor
Ethanol	1.029
Methanol	1.211
Isooctane	0.674
Benzene	0.258
Toluene	0.267
Ethylbenzene	0.284
Xylenes	0.284
Naphthalene	0.207
1-Methylnaphthalene, 2-Methylnaphthalene	0.25

where:

$RRF_1$  = relative response factor of known compound,  
 $RRF_2$  = relative response factor of compound to be determined,  
 $A_1$  = total measured response area of known compound,  
 $A_2$  = total measured response area of compound to be determined,  
 $M_1$  = percent mass of known compound in standard, and  
 $M_2$  = percent mass of compound to be determined in standard.

12.7.2.1 The standard does not have to be limited to two compounds. Any number of compounds may be included, as long as the relative response factor of one of them is known, and all amounts are known relative to the known compound.

12.7.3 Class-based relative response factors may be determined by applying this test method to a standard having known class-based relative concentrations. To do this, follow the method procedure and calculation given in [Sections 14 and 15](#) up to the point where class-based total response areas are obtained (prior to implementing [Eq 5](#)). Class-based relative response factors are obtained from the total class response areas and the known class percent mass concentrations of the standard by applying [Eq 2](#), except in this case all quantities refer to class totals instead of individual compounds.

12.7.3.1 If a standard consisting of known percent mass concentrations of the five hydrocarbon classes of paraffins, isoparaffins, olefins, naphthenes, and aromatics is used, this procedure will leave one of the class RRFs undetermined (whichever one is arbitrary). The relative response factor for the unknown class may be determined by averaging relative response factors of a selection of individual compounds belonging to that class by [Eq 1](#). This is most easily applied to the paraffins class. Then apply [Eq 2](#) using the response areas and relative response factor for paraffins (as  $RRF_1$ ) to determine the other class-based relative response factors.

12.7.3.2 If desired, the calibration procedure may use a standard sample where the percent mass values for each carbon number within each class are known. First, use [Eq 1](#) to calculate the RRF for one of the paraffins relative to methane. Set that paraffin's RRF to  $RRF_1$ , its known percent mass to  $M_1$ , and its measured total area response to  $A_1$  in [Eq 2](#). Calculate each of the remaining compound class RRFs at each carbon number by making the appropriate substitutions for  $M_2$  and  $A_2$  in [Eq 2](#).

### 13. Conditioning

13.1 Before proceeding with measurements after a significant change or maintenance of the GC-VUV system, the procedures in Section 11 should be followed, and a retention time marker list should be generated or verified following the procedure in 12.1 and 12.2.

13.2 In addition, to further prepare the apparatus for automotive spark-ignition fuel measurements, perform the following steps:

13.2.1 Set the GC and detector conditions to measure automotive spark-ignition engine fuel samples. Typical instrument settings are given in Table 5.

13.2.2 Inject 0.3  $\mu\text{L}$  of the solvent test sample defined in 8.4 and run the GC-VUV apparatus through a full oven ramp and cool-down cycle. Repeat two more times.

13.3 If a QC sample as defined in 8.8 has been previously recorded, analyze it using the procedure given in Sections 14 and 15. Otherwise, analyze the system validation mixture defined in 8.7. Verify that group totals for paraffins, isoparaffins, olefins, naphthenes, and aromatics are each within  $\pm 1\%$  by mass of the known totals, and that benzene, toluene, ethylbenzene, total xylenes, and *isooctane* are each within  $\pm 0.5\%$  by mass of their known values.

13.3.1 If the specifications in 13.3 are not met, verify functionality of all GC-VUV components, validity of retention time marker list, and validity/quality of the QC or system validation mixture, or both. Repeat setup methodology in Sections 11, 12, and 13 as necessary to ensure specifications in 13.3 are met before proceeding.

13.3.2 On initial setup or new QC stock, analyze the QC sample and record the measured parameters for comparison with subsequent measurement of the QC sample.

### 14. Procedure

14.1 Inject the sample into the GC injector port. Typical GC method and detector conditions are given in Table 5.

14.2 The system shall record a dark scan immediately after start.

14.3 The system shall record a reference scan immediately after the dark scan.

14.3.1 The reference scan refers to an initial detector scan used as a reference to convert subsequent detector scans to absorbance scans, and is defined in Annex A1. It is not a library reference spectrum.

14.4 The system shall record 125 nm to 240 nm absorbance spectra and time of scan for each detector scan. Conversion of recorded intensity data to absorbance is given in Annex A1.

14.5 At the end of the GC run, the data collection shall automatically stop, and the recorded absorbance spectra processed in order to obtain response areas for each of the hydrocarbon classes and individual compounds being monitored.

14.5.1 Calculate percent mass for each hydrocarbon group and each of the individual compounds/compound groups ethanol, methanol, *isooctane*, benzene, toluene, ethylbenzene, and total xylenes.

14.5.2 (Optional) Calculate percent volume results from the percent mass results and class/compound densities.

14.6 Generate a report displaying the information calculated in 14.5.

### 15. Calculation

NOTE 2—See pertinent information on modeling absorbance data in Annex A2.

15.1 Divide the measured chromatogram into time slices of a given width,  $\Delta t$ . Define the following parameters:

15.1.1 A retention index (RI) window,

15.1.2 A chi-squared iteration threshold, expressed as a percentage,

15.1.3 An  $R^2$  threshold,

15.1.4 A saturation threshold, and

15.1.5 An initial background time region (optional).

15.2 If an initial background time region is defined, calculate a background spectrum from the average of the absorbance scans over the background time region.

15.3 Analyze each time slice using the following algorithm:

15.3.1 Calculate the total absorbance from the sum of the absorbance scans within the time slice.

15.3.1.1 If a background spectrum is defined, subtract the background spectrum from each of the individual absorbance spectra within the time slice. Sum the resulting background-subtracted spectra to obtain the total absorbance spectrum for the time slice.

15.3.1.2 If the absorbance value at a given wavelength exceeds the saturation threshold for any of the absorbance scans within the time slice, remove the data at that wavelength value from the total absorbance and library reference spectra used in subsequent fits for that time slice.

15.3.2 Calculate the average retention index of the time slice using the average elution time of the time slice and the list of retention time markers. A linear interpolation scheme is sufficient.

15.3.3 Construct a list consisting of all compounds in the VUV reference library within  $\pm\text{RI}$  window of the average retention index of the time slice.

**TABLE 5 Typical Instrument Settings for GC-VUV Automotive Spark-Ignition Fuel Measurement**

Column type, dimensions	Capillary, 30 m $\times$ 0.25 mm ID $\times$ 0.25 $\mu\text{m}$ film thickness
Column phase	Nonpolar (for example, 100 % dimethyl polysiloxane)
Injector temperature	250 $^{\circ}\text{C}$
Injection volume	0.3 $\mu\text{L}$
Split ratio	50:1
Column flow	1 mL/min
Oven initial temperature	35 $^{\circ}\text{C}$
Initial hold time	10 min
Oven ramp	7 $^{\circ}\text{C}/\text{min}$
Final oven temperature	200 $^{\circ}\text{C}$
Final hold time	0 min
Detector makeup gas pressure (gauge)	1.72 kPa
Detector scan time	11 ms
Detector averages per scan	20
Detector flow cell temperature	275 $^{\circ}\text{C}$
Transfer tube temperature	275 $^{\circ}\text{C}$

15.3.4 Perform a tiered search on the total absorbance spectrum, drawing from the constructed list of compounds:

15.3.4.1 Construct Eq A2.1 (see Annex A2) assuming a single component contributes to the total absorbance. Select a compound from the list and assign its library reference spectrum to  $A_{1,ref}$  in Eq A2.1. Fit the total absorbance to Eq A2.1 using general linear least squares. Calculate a metric, such as the chi-squared statistic:

$$x^2 = \frac{1}{N} \sum_{i=1}^N \frac{1}{\sigma_i^2} (A_{i,meas} - A_{i,calc})^2 \quad (3)$$

where:

$N$  = the number of data points in an absorbance spectrum fit,

$A_{i,meas}$  = the measured total absorbance at data point  $i$ ,

$A_{i,calc}$  = the calculated total absorbance at data point  $i$ , and

$\sigma_i$  = the uncertainty of measured data point  $i$ , expressed as a standard deviation

If the uncertainty in the measured data have not been estimated, the  $\sigma_i$  may be set to 1. Normalization by the number of data points,  $N$ , is also optional.

15.3.4.2 Repeat the fit for each compound in the list and retain the fit yielding the best chi-square value, along with the best-fit compound's fit value  $f_j$ .

15.3.4.3 Construct Eq A2.1 assuming two compounds contribute to the total absorbance spectrum. Populate  $A_{1,ref}$  and  $A_{2,ref}$  in Eq A2.1 with library reference spectra for each possible pair of compounds from the compound list. Fit the total absorbance to Eq A2.1 for each pair. Retain the pair resulting in the best chi-squared value along with their fit values,  $f_1$  and  $f_2$ . Compare the chi-squared value from the best two-component fit to the chi-squared value from the best one-component fit. If the percent improvement of the chi-squared value for the best two-component fit over the best one-component fit is greater than the chi-squared iteration threshold, retain the two-component result. Otherwise, reject the two-component result and retain the one-component result.

15.3.4.4 Construct Eq A2.1 assuming three compounds contribute to the total absorbance spectrum. Populate  $A_{1,ref}$ ,  $A_{2,ref}$ , and  $A_{3,ref}$  with library reference spectra for each possible triplet of compounds from the compound list. Fit the total absorbance to Eq A2.1 for each triplet. Retain the triplet resulting in the best chi-squared value along with the fit values,  $f_1$ ,  $f_2$ , and  $f_3$ . Compare the chi-squared value from the best three-component fit to the chi-squared value from the best two-component fit. If the percentage improvement of the chi-squared value for the best three-component fit over the best two-component fit is greater than the chi-squared iteration threshold, retain the best three-component result. Otherwise, reject the three-component result and retain the best two-component result, unless the best two-component result was also rejected, in which case retain the best one-component result.

15.3.5 The result of the tiered search procedure is a prediction of the number of compounds that contribute to the total absorbance spectrum, their likely identities, as well as the best-fit values. "Integrate" the library reference spectra of the best-fit compounds by averaging them over the 125 nm to

240 nm region, generating an integration factor for each compound. Multiply the best-fit values,  $f_i$ , by the corresponding integration factors. These are the compounds' contributions to the response area of the time slice.

15.3.6 If the  $R^2$  value, determined from

$$R^2 = 1 - \frac{\sum_{i=1}^N (A_{i,meas} - A_{i,calc})^2}{\sum_{i=1}^N (A_{i,meas} - \bar{A})^2} \quad (4)$$

is less than the  $R^2$  threshold value, reject the analysis results for the time slice (optional). Otherwise, add the compound contributions to the total class response areas according to their class, or to an individual compound's response area if a compound is one of the specified compounds given in Table 4. If an individual compound in Table 4 also belongs to a compound class in Table 3 (for example, *isooctane*), add its response to the individual compound response area and not to the class response area. In Eq 4,  $\bar{A}$  is the wavelength average of the measured total absorbance spectrum.

15.3.7 Iterate the algorithm until all of the time slices have been analyzed.

15.4 Implementation of an analysis criterion for determining whether to analyze a time slice and a background subtraction is permissible. If a background subtraction is used, a criterion for automatically determining that a time region should be used as a background spectrum may be defined.

15.4.1 *Absorbance Check 1*—Compare the change of a response filter over a time slice. If the response filter changes by more than the absorbance threshold, then analyze the time slice. Otherwise, skip the time slice.

15.4.1.1 If a time slice is skipped, the background threshold may be checked and if the response change over the time slice is less than the background threshold, update the background spectrum using the average absorbance spectrum over the time slice.

15.4.2 *Absorbance Check 2*—If the maximum response of the four filters consisting of average 125 nm to 240 nm absorbance, average 170 nm to 200 nm absorbance, average 125 nm to 160 nm absorbance, and average 140 nm to 160 nm absorbance exceeds the maximum response of the same four filters applied to the current background spectrum by more than three times the absorbance threshold, then analyze the time slice (and do not update the background spectrum) regardless of the outcome of Absorbance Check 1.

15.4.3 Other threshold criteria may be used, provided it is first determined that use of alternate threshold criteria does not lessen the accuracy or precision of the test method.

15.5 Due to the similarities of absorbance spectra of compounds belonging to the same class, as well as the similarities of relative response factors among compounds belonging to the same class, it is not necessary to have an explicit representation of all compounds in the VUV reference library. The following substitutions for an uncharacterized compound are permissible and will generally automatically be made by the algorithm:

15.5.1 Library reference spectra of similar compound class and similar carbon number.

15.5.2 Linear combinations of spectra of similar compound class and similar carbon number.

15.6 (Optional): If an  $R^2$  threshold is applied, record the amount of response area rejected by implementation of the  $R^2$  threshold. Compare the rejected amount to the total response area at the end of the analysis. If more than 3 % of the response area was rejected, the analysis should be flagged, and the measurement data and GC-VUV instrumentation should be inspected.

15.7 **Table 6** lists values for analysis parameters used in the statistical study given in Section 17, and are suitable for use with this test method.

15.8 The result of the measurement and analysis procedure are total response areas for each of the hydrocarbon classes and each individually speciated compound. For a given class or specific compound,  $a$ , calculate the percent mass from

$$M_a = 100 \times \frac{A_a \times RRF_a}{\sum_{i=1}^n A_i \times RRF_i} \quad (5)$$

where:

$M_a$  = percent mass for analyte or analyte class  $a$ ,  
 $A_a$  = total response area for analyte or analyte class  $a$ , and  
 $RRF_a$  = relative response factor for analyte or analyte class  $a$ .

The sum runs over all hydrocarbon classes and speciated compounds.

15.8.1 Calculate the total aromatics percent mass by adding the percent mass for the individual mono-aromatics in **Table 4** to the  $C_9+$  aromatics class percent mass.

15.8.2 Calculate total isoparaffins percent mass by adding the percent mass for *isooctane* to the percent mass for the isoparaffins class.

15.9 Calculate total saturate content by summing the percent mass values of the hydrocarbon classes of paraffins, isoparaffins, and naphthenes.

15.10 The percent mass result for analyte or analyte class,  $a$ , may be converted to percent volume using

$$V_a = 100 \times \frac{\frac{M_a}{\rho_a}}{\sum_{i=1}^n \left( \frac{M_i}{\rho_i} \right)} \quad (6)$$

where:

$M_a$  = percent mass for analyte or analyte class  $a$ ,  
 $V_a$  = percent volume for analyte or analyte class  $a$ , and  
 $\rho_a$  = relative liquid density for analyte or average relative density for analyte class  $a$ .

The liquid density values may be obtained from various literature or ASTM publications. For example, densities for many relevant compounds are given in Test Method **D6730** and in ASTM publication DS4A, *Physical Constants of Hydrocarbons C<sup>1</sup> to C<sup>10</sup>*. Average density values may be used for densities of bulk PIONA classes or for PIONA classes at various carbon numbers. Room temperature densities should be used when possible. Recommended densities for use with this test method are given in **Table 7**.

15.10.1 **Eq 6** shall be applied to percent mass values before the individually speciated aromatics percent mass values are added to the  $C_9+$  aromatics percent mass, and before the *isooctane* percent mass value is added to the isoparaffins percent mass. After the percent volume values are calculated, the percent volume values of the mono-aromatics from **Table 4** shall be added to the percent volume result for the  $C_9+$  aromatics class in order to report total aromatics content. Similarly for *isooctane* and the isoparaffins class.

15.11 Calculate total saturate percent volume by summing the percent volume values of the hydrocarbon classes of paraffins, isoparaffins, and naphthenes.

## 16. Report

16.1 Report percent mass and, optionally, percent volume for each of the compound classes of paraffins, isoparaffins, olefins, naphthenes, aromatics, and total saturates to the nearest 0.1 %. Report percent mass and, optionally, percent volume for each of the individually speciated compounds and compound groups in **Table 4** to the nearest 0.01 %.

**TABLE 6 Parameters Used in Analysis of GC-VUV Scan Data**

Parameter	Value
Time slice width	0.02 min
RI window	±25
Initial background region	1.8 min to 2.0 min
Saturation threshold	1.0 AU
Absorbance threshold	0.001 AU
Background threshold	0.0003 AU
Response filter to apply Absorbance Check 1	Average 140 nm to 160 nm absorbance
Response filter to apply background check	Average 140 nm to 160 nm absorbance
Response filter(s) to apply Absorbance Check 2	Average 125 nm to 240 nm absorbance Average 170 nm to 200 nm absorbance Average 125 nm to 160 nm absorbance Average 140 nm to 160 nm absorbance
Chi-square iteration threshold	40 %



**TABLE 7 Relative Densities for Various Hydrocarbon Classes and Individual Compounds**

Hydrocarbon Class/Compound	Relative Density
Paraffin	0.660
Isoparaffin	0.660
Olefin	0.657
Naphthene	0.774
C9+ Aromatics	0.872
Ethanol	0.789
Methanol	0.792
<i>Is</i> ooctane	0.660
Benzene	0.879
Toluene	0.867
Ethylbenzene	0.867
Xylenes	0.870
Naphthalene	1.025
Methylnaphthalenes	1.020

**TABLE 8 Percent Mass Values for Samples Measured in Repeatability Tests**

Hydrocarbon Group/Species	Sample 1	Sample 2	Sample 3	Sample 4
Paraffins	12.1	6.0	9.7	10.1
Isoparaffins	53.4	36.8	46.4	35.5
Olefins	5.7	9.6	2.5	15.0
Naphthenes	2.7	4.1	11.9	7.1
Aromatics	16.9	32.8	28.7	23.1
Ethanol	8.93	10.48	0.00	8.73
<i>Is</i> ooctane	22.26	6.57	6.67	4.06
Naphthalene	0.12	0.14	0.31	0.24
Methylnaphthalenes	0.16	0.06	0.54	0.22
Benzene	0.32	0.32	0.72	0.62
Toluene	1.75	2.63	4.95	4.19
Ethylbenzene	0.28	0.52	1.37	1.05
Xylenes	1.56	4.56	7.36	6.11
Total Saturates	68.2	46.9	68.0	52.6

16.1.1 (Optional): Add the percent mass/percent volume values of naphthalene and 1- and 2- methylnaphthalene to the total aromatics percent mass/percent volume.

## 17. Precision and Bias

17.1 The repeatability and reproducibility will ultimately be determined by an interlaboratory study<sup>5</sup> in accordance with ASTM methodology. This section reports temporary, single-laboratory repeatability standard deviations for the quantities listed in [Table 8](#).

17.1.1 Repeatability tests were carried out by a single laboratory on four ASTM gasoline proficiency samples. In order to mitigate effects of sample change during repeated measurement, each sample was split into four vials. Each vial was measured three times, for a total of twelve measurements

<sup>5</sup> An interlaboratory study of this test method is being conducted and a complete precision statement is expected to be available on or before 2021.

per sample. A total of 14 parameters were determined during each measurement: group paraffin, isoparaffin, olefin, naphthene, aromatic, and total saturate concentrations, in addition to specific concentrations of ethanol, *is*ooctane, benzene, toluene, ethylbenzene, total xylenes, naphthalene, and total methylnaphthalenes. [Table 8](#) shows the percent mass values of the various parameters for the four samples. The standard deviation was calculated for 13 of the 14 parameters in accordance with section 6.2.1 of Practice [D6300](#), and the results are given in [Table 9](#). A separate repeatability standard deviation was not determined for total saturates since it is determined completely from other measured parameters.

## 18. Keywords

18.1 gas chromatography; gasoline analysis; PIONA analysis; spark-ignition engine fuel analysis; vacuum ultraviolet absorption spectroscopy

**TABLE 9 Single Laboratory Percent Mass Repeatability Standard Deviations, Determined According to Practice D6300 Section 6.2.1**

Hydrocarbon Group/Species	Sample 1	Sample 2	Sample 3	Sample 4
Paraffins	0.14	0.05	0.05	0.06
Isoparaffins	0.14	0.13	0.18	0.34
Olefins	0.06	0.13	0.10	0.26
Naphthenes			0.16	
Aromatics	0.06	0.15	0.06	0.13
Ethanol	0.033	0.026	0.000	0.040
Isooctane	0.105	0.027	0.050	0.036
Benzene	0.002	0.003	0.004	0.005
Toluene	0.006	0.009	0.013	0.026
Ethylbenzene	0.003	0.003	0.007	0.006
m,p,o - Xylene	0.006	0.017	0.040	0.045
Naphthalene	0.001	0.001	0.001	0.002
Methylnaphthalenes			0.003	

## ANNEXES

### (Mandatory Information)

#### A1. GENERATING ABSORBANCE SPECTRA FROM INTENSITY SCAN DATA

A1.1 The following converts recorded scan intensity data to VUV absorbance spectra, assuming the availability of a previously recorded dark intensity scan,  $I_d(\lambda)$ , and reference intensity scan,  $I_r(\lambda)$ .

A1.1.1 The dark scan is collected at the beginning of the chromatogram with the lamp shutter closed. The dark scan is subtracted from all subsequent intensity scans. The reference scan is collected at the beginning of the chromatogram with the shutter open, but with only carrier/makeup gas flowing through the flow cell.

A1.1.2 After collecting dark and reference scans, the detector continues to collect intensity scans,  $I(\lambda)$ , at regular intervals. The detector array typically has settings for scan time, in ms, and number of averages per scan. The total time per scan is the scan time multiplied by the number of averages. For example, a scan time of 11 ms with 20 averages results in a scan rate of about 4.5 scans/s.

A1.1.3 The absorbance is given by

$$A(\lambda) = \log_{10}\left(\frac{1}{\tau(\lambda)}\right) = \log_{10}\left(\frac{I_r(\lambda) - I_d(\lambda)}{I(\lambda) - I_d(\lambda)}\right) \quad (\text{A1.1})$$

#### A2. MODELING ABSORBANCE DATA

A2.1 The separation of automotive spark-ignition fuel samples will typically involve multiple cases of coelution, where more than one analyte compound can exist in the flow cell at any given time. The absorbance for a given number of compounds  $n$  can be written as

$$A = \sum_{i=1}^n f_i A_{i,ref} = (f_1 A_{1,ref} + f_2 A_{2,ref} + \dots + f_n A_{n,ref}) \quad (\text{A2.1})$$

where:

$A_{i,ref}$  = a reference absorbance spectrum for molecule  $i$ , and

$f_i$  = a weighting factor related to the amount of compound  $i$  present.

A2.2 The absorbances of multiple scans are summed over a time region, or “time slice.” The relationship between this total absorbance and the individual constituents is also given by Eq A2.1, which then includes one term for each compound that elutes within the time slice.

A2.3 A measured absorbance can be fit using Eq A2.1 and a general linear least squares optimization procedure.<sup>6</sup> The  $f_i$  are the parameters to be fit. Each of the reference spectra can be “integrated” using the same integration filter that generated the chromatogram response (in this case by averaging over 125 nm to 240 nm), resulting in an integration factor for each reference spectrum included in Eq A2.1. The fit  $f_i$  multiplied by the corresponding integration factors are the contributions from each of the coeluting compounds to the chromatogram response within the time slice.

<sup>6</sup> Press, W. H., Flannery, B. P., Teukolsky, S. A., and Vetterling, W. T., *Numerical Recipes in C: The Art of Scientific Computing, Second Edition*, Cambridge University Press, Cambridge, UK, 1992.

## APPENDIX

## (Nonmandatory Information)

**X1. DETERMINATION OF CARBON NUMBER DISTRIBUTION OF PARAFFINS, ISOPARAFFINS, OLEFINS, NAPHTHENES, AND AROMATICS**

X1.1 This test method may be extended to provide carbon number percent mass or percent volume distributions, or both, of the five hydrocarbon classes of paraffins, isoparaffins, olefins, naphthenes, and aromatics.

X1.1.1 Each class response area is further broken down into carbon number—in 15.3.6, add the compound contributions to the total class response areas according to their class and carbon number stored in the VUV library, or to an individual compound's response area if a compound is one of the speciated compounds given in Table 4.

X1.1.2 In 15.8, apply Eq 5 to each speciated compound and each analyte class for each carbon number. The sum in Eq 5 runs over each speciated compound in Table 4, and each carbon number grouping for each class in Table 3.

X1.1.2.1 Use class-based relative response factors in Table 3 for all carbon numbers of a given class.

X1.1.2.2 Alternatively, class relative response factors may be further broken down into carbon number-specific relative response factors using, for example, the procedure in 12.7.3.2.

X1.1.3 Combine the percent mass for each carbon number with the percent mass of any speciated compound from Table 4 that belongs to that carbon number and class. For example, add the *isooctane* percent mass to the C<sub>8</sub> isoparaffins percent mass to obtain the total C<sub>8</sub> isoparaffins percent mass.

X1.1.4 Combine the percent mass for all carbon numbers of a given class to obtain the class total percent mass.

X1.1.5 Use Eq 6 to convert percent mass values to percent volume values. Apply Eq 6 before combining percent mass values in X1.1.3 and X1.1.4.

X1.1.5.1 Use average class density values from Table 7 for all carbon numbers of a given class.

X1.1.5.2 Alternatively, define average density values for each carbon number of each class for use in Eq 6.

X1.1.6 Combine the percent volume for each carbon number with the percent volume of any speciated compound from Table 4 that belongs to that carbon number and class. For example, add the *isooctane* percent volume to the C<sub>8</sub> isoparaffins percent volume to obtain the total C<sub>8</sub> isoparaffins percent volume.

X1.1.7 Combine the percent volume for all carbon numbers of a given class to obtain the class total percent volume.

X1.1.8 In addition to the reporting items in Section 16, report percent mass and, optionally, percent volume for each carbon number of each class to the nearest 0.01 %.

X1.1.8.1 (Optional): Add the percent mass/percent volume of naphthalene to the percent mass/percent volume of C<sub>10</sub> aromatics and to total aromatics. Add the percent mass/percent volume of 1- and 2-methylnaphthalene to the percent mass/percent volume of C<sub>11</sub> aromatics and total aromatics.

X1.1.9 Figs. X1.1-X1.5 show comparisons of the carbon number distributions of the repeatability samples 1 through 4, using the response factors from Table 3 and Table 4.

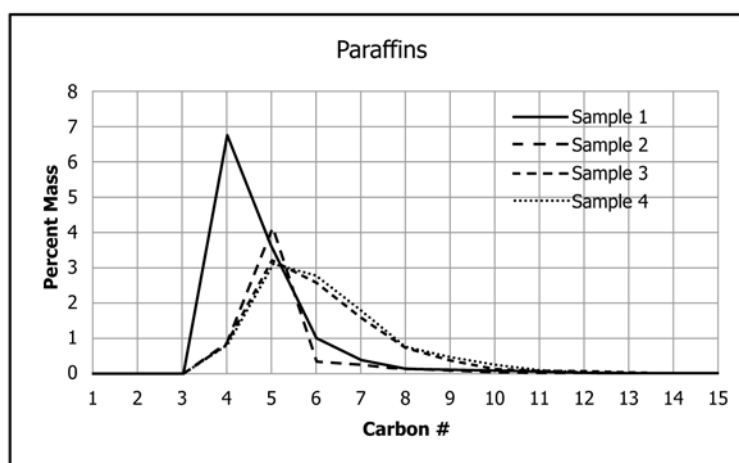


FIG. X1.1 Comparison of Carbon Number Distributions of Paraffins for Repeatability Samples 1 through 4

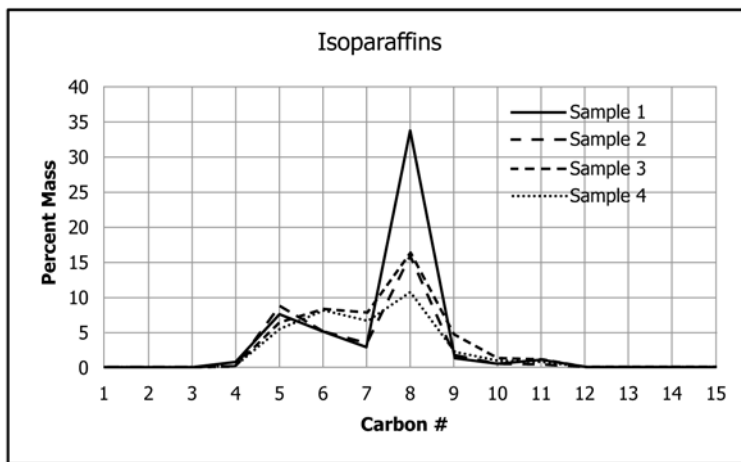


FIG. X1.2 Comparison of Carbon Number Distributions of Isoparaffins for Repeatability Samples 1 through 4

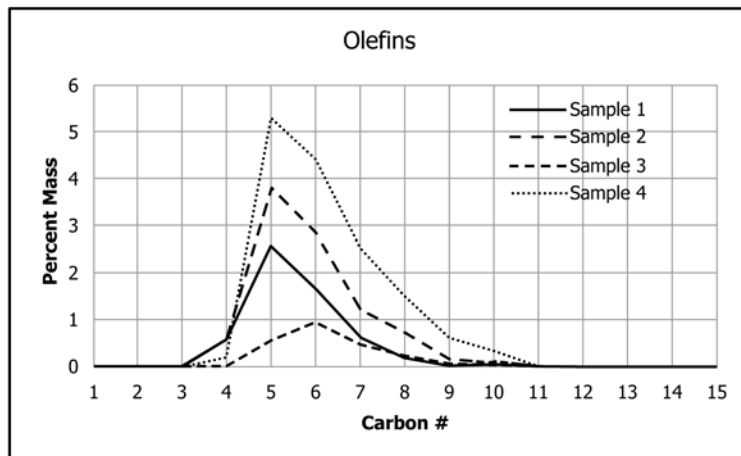


FIG. X1.3 Comparison of Carbon Number Distributions of Olefins for Repeatability Samples 1 through 4

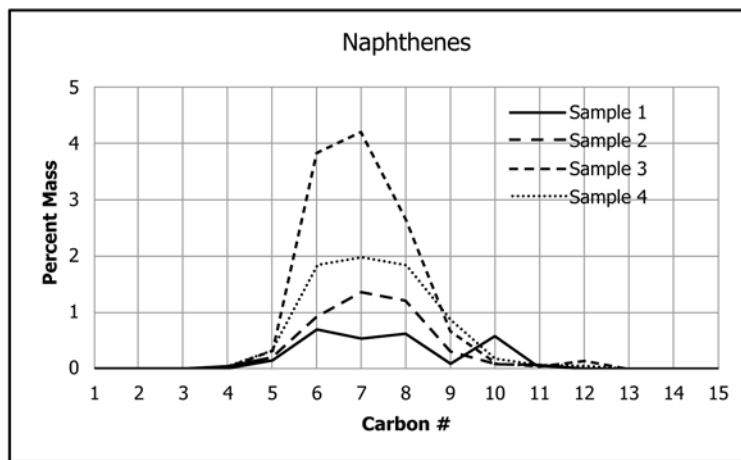


FIG. X1.4 Comparison of Carbon Number Distributions of Naphthenes for Repeatability Samples 1 through 4

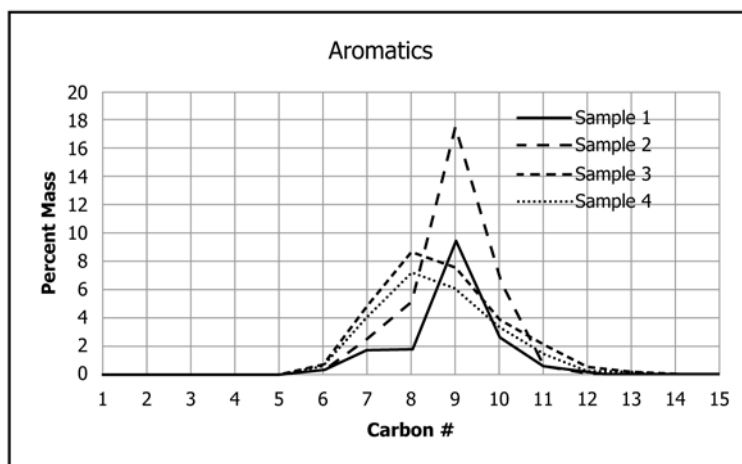


FIG. X1.5 Comparison of Carbon Number Distributions of Aromatics for Repeatability Samples 1 through 4

### RELATED MATERIAL

ASTM Test Method D1319 for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption  
 ASTM Test Method D3606 for Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography  
 ASTM Test Method D4815 for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography  
 ASTM Test Method D5580 for Determination of Benzene, Toluene, Ethylbenzene, p/m-Xylene, o-Xylene, C9 and Heavier Aromatics, and Total Aromatics in Finished Gasoline by Gas Chromatography  
 ASTM Test Method D5599 for Determination of Oxygenates in Gasoline

by Gas Chromatography and Oxygen Selective Flame Ionization Detection  
 ASTM Test Method D5769 for Determination of Benzene, Toluene, and Total Aromatics in Finished Gasolines by Gas Chromatography/Mass Spectrometry  
 ASTM Test Method D5845 for Determination of MTBE, ETBE, TAME, DIPE, Methanol, Ethanol and tert-Butanol in Gasoline by Infrared Spectroscopy  
 ASTM Test Method D6550 for Determination of Olefin Content of Gasolines by Supercritical-Fluid Chromatography

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