Designation: D6591 – 19

Designation: 548/06

Standard Test Method for Determination of Aromatic Hydrocarbon Types in Middle Distillates—High Performance Liquid Chromatography Method with Refractive Index Detection[1]

This standard is issued under the fixed designation D6591; 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.

INTRODUCTION

This test method has the same title as IP 548-06 and is intended to be technically equivalent. The ASTM format for test methods has been used, and where possible, equivalent ASTM test methods have replaced the IP or ISO standards.

The test method is intended to be used as one of several possible alternative instrumental test methods that are aimed at quantitative determination of hydrocarbon types in fuels. This does not imply that a correlation necessarily exists between this and any other test method intended to give this information, and it is the responsibility of the user to determine such correlation if necessary.

1. Scope*

1.1 This test method covers a high performance liquid chromatographic test method for the determination of mono-aromatic, di-aromatic, tri+-aromatic, and polycyclic aromatic hydrocarbon contents in diesel fuels and petroleum distillates boiling in the range from 150 °C to 400 °C. The total aromatic content in % m/m is calculated from the sum of the corresponding individual aromatic hydrocarbon types.

NOTE 1—Aviation fuels and petroleum distillates with a boiling point range from 50 °C to 300 °C are not determined by this test method and should be analyzed by Test Method D6379 or other suitable equivalent test methods.

1.2 The precision of this test method has been established for diesel fuels and their blending components, containing from 4 % to 40 % (m/m) mono-aromatic hydrocarbons, 0 % to 20 % (m/m) di-aromatic hydrocarbons, 0 % to 6 % (m/m) tri+-aromatic hydrocarbons, 0 % to 26 % (m/m) polycyclic aromatic hydrocarbons, and 4 % to 65 % (m/m) total aromatic hydrocarbons.

1.3 Compounds containing sulfur, nitrogen, and oxygen are possible interferents. Mono-alkenes do not interfere, but conjugated di- and poly-alkenes, if present, are possible interferents.

1.4 By convention, this standard defines the aromatic hydrocarbon types on the basis of their elution characteristics from the specified liquid chromatography column relative to model aromatic compounds. Quantification is by external calibration using a single aromatic compound, which may or may not be representative of the aromatics in the sample, for each aromatic hydrocarbon type. Alternative techniques and methods may classify and quantify individual aromatic hydrocarbon types differently.

1.5 Fatty Acid Methyl Esters (FAME), if present, interfere with tri+-aromatic hydrocarbons. If this method is used for diesel containing FAME, the amount of tri+-aromatics will be over estimated.

1.6 This test method includes a Relative Bias section for Test Method D6591 versus Test Method D1319 and Test Method D5186 versus Test Method D6591 for diesel fuels only. The applicable concentration ranges of the correlations are presented in the Relative Bias section. The correlations are applicable only in the stated ranges.

1.7 This test method and correlations were developed for diesel samples not containing biodiesel; the presence of biodiesel will interfere with the results. The correlation equations are only applicable between these concentration ranges and to diesel fuels that do not contain biodiesel.

[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 D02.04.0C on Liquid Chromatography.

This test method is based on material published in *IP Standard Methods for Analysis and Testing of Petroleum and Related Products and British Standard 2000 Parts*, copyright The Institute of Petroleum, 61 New Cavendish Street, London W1M 8AR. Adapted with permission of The Institute of Petroleum.

Current edition approved June 1, 2019. Published August 2019. Originally approved in 2000. Last previous edition approved in 2018 as D6591 – 18. DOI: 10.1520/D6591-19.

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

1.8 *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.*

1.9 *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]

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

D2425 Test Method for Hydrocarbon Types in Middle Distillates by Mass Spectrometry

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5186 Test Method for Determination of the Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels By Supercritical Fluid Chromatography

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

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

2.2 *Energy Institute Standard:*[3]

IP 548 Test Method for Determination of Aromatic Hydrocarbon Types in Middle Distillates – High Performance Liquid Chromatography Method with Refractive Index Detection

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *di-aromatic hydrocarbons (DAHs), n*—in this test method, compounds that have a longer retention time on the specified polar column than the majority of mono-aromatic hydrocarbons, but a shorter retention time than the majority of tri+-aromatic hydrocarbons.

3.1.2 *mono-aromatic hydrocarbons (MAHs), n*—in this test method, compounds that have a longer retention time on the specified polar column than the majority of non-aromatic hydrocarbons but a shorter retention time than the majority of DAHs.

3.1.3 *non-aromatic hydrocarbons, n*—in this test method, compounds that have a shorter retention time on the specified polar column than the majority of mono-aromatic hydrocarbons.

3.1.4 *polycyclic aromatic hydrocarbons (POLY-AHs), n*—in this test method, sum of the di-aromatic hydrocarbons and tri+-aromatic hydrocarbons.

3.1.5 *total aromatic hydrocarbons, n*—in this test method, sum of the MAHs, DAHs, and T+AHs.

3.1.6 *tri+-aromatic hydrocarbons (T+AHs), n*—in this test method, compounds that have a longer retention time on the specified polar column than the majority of DAHs.

3.1.6.1 *Discussion*—The elution characteristics of aromatic and non-aromatic compounds on the specified polar column have not been specifically determined for this test method. Published and unpublished data indicate the major constituents for each hydrocarbon type as follows: *(1)* non-aromatic hydrocarbons: acyclic and cyclic alkanes (paraffins and naphthenes), mono-alkenes (if present), *(2)* MAHs: benzenes, tetralins, indanes, thiophenes, and conjugated poly-alkenes, *(3)* DAHs: naphthalenes, biphenyls, indenes, fluorenes, acenaphthenes, and benzothiophenes and dibenzothiophenes, *(4)* T+AHs: phenanthrenes, pyrenes, fluoranthenes, chrysenes, triphenylenes, and benzanthracenes.

4. Summary of Test Method

4.1 A known mass of sample is diluted in the mobile phase, and a fixed volume of this solution is injected into a high performance liquid chromatograph, fitted with a polar column. This column has little affinity for the non-aromatic hydrocarbons while exhibiting a pronounced selectivity for aromatic hydrocarbons. As a result of this selectivity, the aromatic hydrocarbons are separated from the non-aromatic hydrocarbons into distinct bands in accordance with their ring structure, that is, MAHs, DAHs, and T+AHs. At a predetermined time, after the elution of the DAHs, the column is backflushed to elute the T+AHs as a single sharp band.

4.2 The column is connected to a refractive index detector that detects the components as they elute from the column. The electronic signal from the detector is continually monitored by a data processor. The amplitudes of the signals (peak areas) from the sample aromatics are compared with those obtained from previously measured calibration standards in order to calculate percent m/m MAHs, DAHs, and T+AHs in the sample. The sum of the percentages by mass of DAHs and T+AHs is reported as the percent m/m POLY-AH. The sum of MAHs, DAHs, and T+AHs is reported as the total aromatic content (percent m/m) of the sample.

5. Significance and Use

5.1 The aromatic hydrocarbon content of motor diesel fuel is a factor that can affect exhaust emissions and fuel combustion characteristics, as measured by cetane number.

5.2 The United States Environmental Protection Agency (US EPA) regulates the aromatic content of diesel fuels. California Air Resources Board (CARB) regulations place limits on the total aromatics content and polynuclear aromatic

[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] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.

hydrocarbon content of motor diesel fuel, thus requiring an appropriate analytical determination to ensure compliance with the regulations.

5.3 This test method is applicable to materials in the same boiling range as motor diesel fuels and is unaffected by fuel coloration. Test Method D1319, which has been mandated by the US EPA for the determination of aromatics in motor diesel fuel, excludes materials with final boiling points greater than 315 °C (600 °F) from its scope. Test Method D2425 is applicable to the determination of both total aromatics and polynuclear aromatic hydrocarbons in diesel fuel, but is much more costly and time consuming to perform. Test Method D5186, currently specified by CARB, is also applicable to the determination of both total aromatics and polynuclear aromatic hydrocarbons in diesel fuel. Test Method D5186, however, specifies the use of supercritical fluid chromatography equipment that may not be readily available.

NOTE 2—Test Method D5186 was previously specified by CARB as an alternative to Test Method D1319.

6. Apparatus

6.1 *High Performance Liquid Chromatograph (HPLC)*—Any HPLC capable of pumping the mobile phase at flow rates between 0.5 mL/min and 1.5 mL/min, with a precision better than 0.5 % and a pulsation of <1 % full-scale deflection under the test conditions described in Section 9.

6.2 *Sample Injection System,* capable of injecting 10 µL (nominal) of sample solution with a repeatability better than 1 %.

6.2.1 An equal and constant volume of the calibration and sample solutions shall be injected into the chromatograph. Both manual and automatic sample injection systems (using either complete or partial filling of the sample loop) will, when used correctly, meet the repeatability requirements laid down in 6.2. When using the partial loop-filling mode, it is recommended that the injection volume should be less than half the total loop volume. For complete filling of the loop, best results are obtained by overfilling the loop at least six times.

NOTE 3—The repeatability of the injection system may be checked by comparing peak areas from at least four injections of the system performance standard (see 9.3).

6.2.2 Sample and calibration injection volumes other than 10 µL (typically in the range from 3 µL to 20 µL) may be used, provided they meet the requirements laid down for injection repeatability (see 6.2), refractive index sensitivity and linearity (see 9.4.2 and 10.1.5), and column resolution (see 9.4.3).

6.3 *Sample Filter,* if required (see 10.2.1)—A microfilter of porosity 0.45 µm or less, which is chemically-inert towards hydrocarbon solvents, is recommended for the removal of particulate matter from the sample solutions.

6.4 *Column System*—Any stainless steel HPLC column(s) packed with an approved amino-bonded (or polar amino/cyano-bonded) silica stationary phase is suitable, provided it meets the resolution requirements laid down in 9.4.3. See Appendix X1 for guidance on the selection and use of suitable column systems.

6.5 *HPLC Column Oven*—Any suitable HPLC column oven (block heating or air circulating) capable of maintaining a constant temperature (±1 °C) within the range from 20 °C to 40 °C.

NOTE 4—The refractive index detector is sensitive to both sudden and gradual changes in the temperature of the eluent. All necessary precautions should be taken to establish constant temperature conditions throughout the liquid chromatograph system. The temperature should be optimized depending on the stationary phase.

NOTE 5—Alternative forms of temperature control, for example, temperature-controlled laboratories, are permitted.

6.6 *Backflush Valve*—Any manual or automatic (air or electrically actuated) flow-switching valve designed for use in HPLC systems that is capable of operating at pressures up to 2×10^4 kPa.

6.7 *Refractive Index Detector*—Any refractive index detector may be used provided it is capable of being operated over the refractive index range from 1.3 to 1.6, meets the sensitivity requirement specified in 9.4.2, gives a linear response over the calibration range, and has a suitable output signal for the data system.

NOTE 6—If the refractive index detector has a facility for independent temperature control, it is recommended that this be set at the same temperature as the column oven.

6.8 *Computer or Computing Integrator*—Any data system can be used provided it is compatible with the refractive index detector, has a minimum sampling rate of 1 Hz, and is capable of peak area and retention time measurement. The data system should also have minimum facilities for post-analysis data processing, such as baseline correction and reintegration. The ability to perform automatic peak detection and identification and to calculate sample concentrations from peak area measurements is recommended but not essential.

6.9 *Volumetric Flasks,* Grade B or better, of 10 mL and 100 mL capacity.

6.10 *Analytical Balance,* accurate to ±0.0001 g.

7. Reagents

7.1 *Cyclohexane,* >99 % pure.

NOTE 7—Cyclohexane may contain benzene as an impurity.

7.2 *Heptane,* HPLC Grade. For use as HPLC mobile phase. (**Warning—**Heptane is highly flammable and may cause irritation by inhalation, ingestion, or skin contact.)

NOTE 8—Batch-to-batch variation of the solvent quality in terms of water content, viscosity, refractive index, and purity could cause unpredictable column behavior. Drying and filtering the mobile phase could help to reduce the effect of the trace impurities in the solvent.

NOTE 9—It is recommended practice to de-gas the HPLC mobile phase before use; this can be done conveniently, on-line, or off-line by helium sparging, vacuum de-gassing, or ultrasonic agitation. A failure to de-gas the mobile phase may lead to negative peaks.

7.3 *o-Xylene (1,2-Dimethylbenzene),* ≥98 % pure.

7.4 *1-Methylnaphthalene,* ≥98 % pure.

7.5 *Phenanthrene,* ≥98 % pure.

7.6 *Dibenzothiophene,* ≥95 % pure.

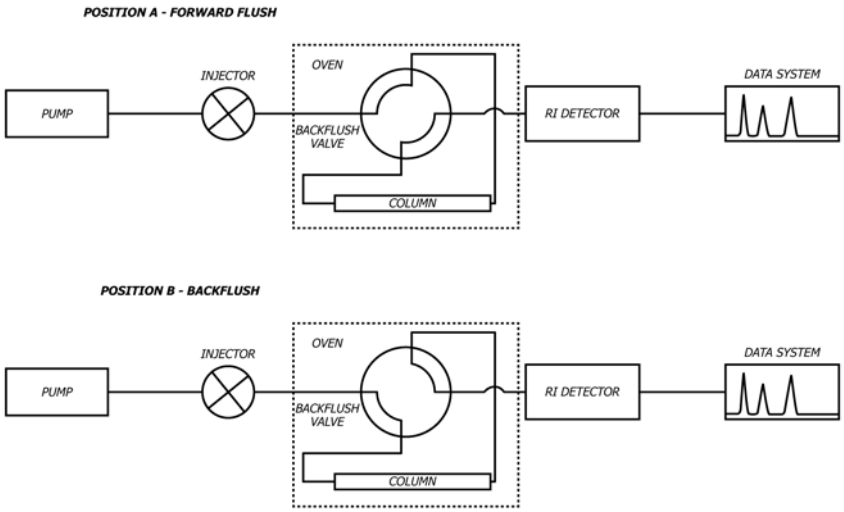

FIG. 1 Diagrammatic Representation of Liquid Chromatograph

7.7 *9-Methylanthracene,* ≥95 % pure. (**Warning—**Gloves should be worn when handling aromatic compounds (for example, disposable vinyl gloves).)

NOTE 10—Purity is determined by gas chromatography with flame ionization detection. The highest purity standards available should be used.

8. Sampling

8.1 Unless otherwise specified in the commodity specification, samples are taken by following Practice D4057 or D4177, or a similar standard. In certain situations, sampling is done in accordance with the requirements of national standards or regulations for the sampling of the product under test.

9. Apparatus Preparation

9.1 Set up the chromatograph, injection system, column, backflush valve, column oven, refractive index detector, and computing integrator in accordance with the appropriate equipment manuals. Install the HPLC column and backflush valve in the column oven. Insert the backflush valve so that the detector is always connected independently of the direction of flow through the column (see Fig. 1). Maintain the sample injection valve at the same temperature as the sample solution; in most cases this will be at room temperature.

NOTE 11—The column oven is optional if alternative arrangements are made to maintain a constant temperature environment, for example, a temperature-controlled laboratory (see 6.5). It is recommended to install the backflush valve in the column oven and to install the apparatus away from drafts (that is, not near air-conditioning unit or fume cupboard). Pipework and/or valving which is not temperature controlled should be insulated.

NOTE 12—Regular maintenance of the liquid chromatograph and its components is important to ensure consistent performance. Leakages and partial blockage of filters, frits, injector needles, and valve rotors can produce flow rate inconsistencies and poor injector performance.

9.2 Adjust the flow rate of the mobile phase to a constant 1.0 mL⁄min ± 0.2 mL⁄min, and ensure the reference cell of the refractive index detector is full of mobile phase. Allow the temperature of the column oven (and refractive index detector, if equipped with temperature control) to stabilize.

9.2.1 To minimize drift, it is essential to make sure the reference cell is full of solvent. The best way to accomplish this is either *(1)* to flush the mobile phase through the reference cell (then isolate the reference cell to prevent evaporation of the solvent) immediately prior to analysis, or *(2)* to continuously make up for solvent evaporation by supplying a steady flow through the reference cell. The make-up flow is optimized so that reference and analytical cell mis-match due to drying-out, temperature, or pressure gradients are minimized. Typically, this can be accomplished with a make-up flow set at one tenth of the analytical flow.

NOTE 13—The flow rate may be adjusted (typically within the range from 0.8 mL⁄min to 1.2 mL⁄min) to an optimum value in order to meet the resolution requirements specified in 9.4.3.

9.3 Prepare a system performance standard (SPS) by weighing cyclohexane (1.0 g ± 0.1 g), *o*-xylene (0.5 g ± 0.05 g), dibenzothiophene (0.05 g ± 0.005 g), and 9-methylanthracene (0.05 g ± 0.005 g) into a 100 mL volumetric flask and making up to the mark with heptane. Ensure that the dibenzothiophene and 9-methylanthracene are dissolved in the *o*-xylene-cyclohexane mixture (for example, by using an ultrasonic bath) before adding heptane.

NOTE 14—The SPS may be kept for up to one year if stored in a tightly stoppered bottle in a dark place between 5 °C and 25 °C.

9.4 When operating conditions are steady, as indicated by a stable horizontal baseline, inject 10 µL of the SPS (see 9.3) and record the chromatogram, using the data system. Ensure the baseline drift over the period of the HPLC analysis run is less than 0.5 % of the peak height for cyclohexane.

NOTE 15—A baseline drift greater than this indicates problems with the temperature control of the column/refractive index detector or polar material eluting from the column, or both. A period of up to 1 h may be required before the liquid chromatograph reaches steady-state conditions.

9.4.1 Ensure that baseline separation is obtained between all four components of the SPS (see Fig. 2).

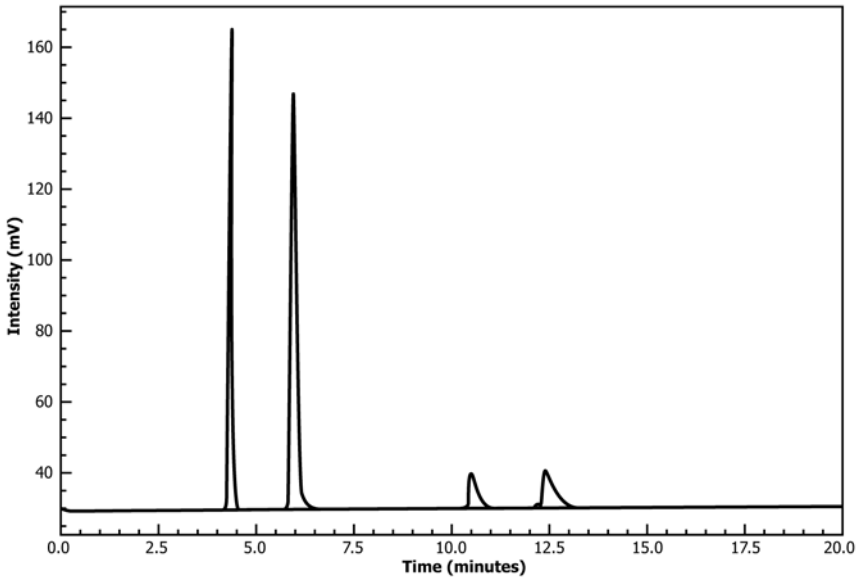

FIG. 2 Chromatogram of System Calibration Standard

9.4.2 Ensure that the data system can accurately measure the peak areas of dibenzothiophene and 9-methylanthracene.

NOTE 16—The S/N (signal-to-noise) ratio for dibenzothiophene and 9-methylanthracene should be 3:1 or greater.

9.4.3 Ensure that the resolution between cyclohexane and *o*-xylene is not less than 5.0.

9.4.3.1 *Column Resolution*—Calculate the resolution between cyclohexane and *o*-xylene as follows:

$$\text{Resolution} = \frac{2 \times (t_2 - t_1)}{1.699 \times (y_1 + y_2)} \quad (1)$$

where:

t_1 = retention time of cyclohexane peak in seconds,
t_2 = retention time of *o*-xylene peak in seconds,
y_1 = half-height peak width of cyclohexane in seconds, and
y_2 = half-height peak width of *o*-xylene in seconds.

If the resolution is less than 5.0, check to see that all system components are functioning correctly and that the chromatographic dead volume has been minimized. Adjust the flow rate to see if this improves the resolution, and make sure the mobile phase is of sufficiently high quality. Finally, regenerate or replace the column.

9.5 Measure the retention times of the dibenzothiophene and 9-methylanthracene peaks, using the data system.

9.6 Calculate the backflush time, *B*, in seconds, using the following equation:

$$B = t_A + 0.4(t_B - t_A) \quad (2)$$

where:

t_A = retention time of dibenzothiophene in seconds, and
t_B = retention time of 9-methylanthracene in seconds.

NOTE 17—The backflush time is the time after injection at which the backflush valve will be actuated in order to elute T+AHs as a single sharp peak.

9.7 When operating conditions are steady, as indicated by a stable horizontal baseline, inject 10 µL of the SPS (see 9.3) and record the chromatogram, using the data system. Actuate the backflush valve at the predetermined time (see 9.6) to elute the T+AHs as a single sharp peak (see Fig. 3). When the analysis is finished, reverse the flow direction of the middle phase (that is, return to forward flush) and allow the baseline to stabilize before the next injection.

9.8 Repeat 9.7, and ensure that the repeatabilities for peak area measurements of *o*-xylene, dibenzothiophene, and 9-methylanthracene are within the precision of this test method.

NOTE 18—If peak area repeatabilities are poor, check to see that the injection system is working optimally and that the baseline is stable (minimal drift) and noise-free.

10. Procedure

10.1 *Calibration:*

10.1.1 Prepare four calibration standards (*A, B, C,* and *D*), at the approximate concentrations given in Table 1, by weighing, to the nearest 0.0001 g, the appropriate materials into 100 mL volumetric flasks and making up to the mark with heptane.

NOTE 19—The recommended concentrations in Table 1 will cover most petroleum materials distilling in the diesel boiling range. Other standard concentrations may be used, provided they meet the requirements of the test method (that is, linearity, detector sensitivity, and column resolution).

NOTE 20—The calibration standard solutions should be stored in tightly stoppered bottles (for example, 10 mL volumetric flasks) in a dark place between 5 °C and 25 °C. Under these conditions, the solutions are viable for at least six months.

10.1.2 When operating conditions are steady (see 9.4), inject 10 µL of calibration standard *A*. Record the chromatogram, and measure the peak areas for each aromatic standard (see Fig. 3). Actuate the backflush valve at the predetermined time (see 9.6) to elute the T+AH standard as a single sharp peak. When the analysis is finished, reverse the flow direction of the mobile phase (that is, return to forward flush) and allow the baseline to stabilize before the next injection.

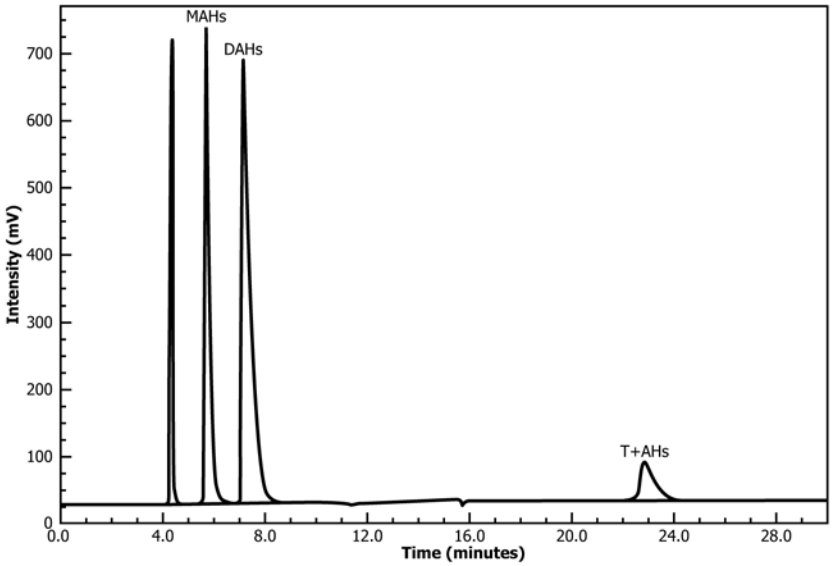

FIG. 3 Chromatogram of Calibration Standard A

TABLE 1 Concentrations of Calibration Components

Calibration Standard	Cyclohexane g/100 mL	*o*-Xylene g/100 mL	1-Methyl-naphthalene g/100mL	Phenanthrene g/100mL
A	5.0	4.0	4.0	0.4
B	2.0	1.0	1.0	0.2
C	0.5	0.25	0.25	0.05
D	0.1	0.05	0.02	0.01

10.1.3 Repeat 10.1.2 using calibration standards *B, C,* and *D*.

10.1.4 If the peak area for phenanthrene in calibration standard *D* is too small to measure accurately, prepare a new calibration standard *D* with a higher concentration of phenanthrene (for example, 0.02 g/100 mL) and proceed in accordance with 10.1.1.

10.1.5 Plot percent m/v (g/100 mL) concentration against area counts for each aromatic standard, that is, *o*-xylene, 1-methylnaphthalene, and phenanthrene. Calibration plots shall be linear with a correlation coefficient greater than 0.999 and an intercept of less than ±0.01 g/100 mL.

NOTE 21—A computer or a data system may be used to interpret these calibrations.

NOTE 22—It should only be necessary to calibrate the refractive index detector on a daily basis.

NOTE 23—It is recommended that a reference diesel or one of the four calibration standards be run after every five samples to check the stability of the system.

10.2 *Analysis of Samples:*

10.2.1 Weigh, to the nearest 0.001 g, between 0.9 g and 1.1 g of sample into a 10 mL volumetric flask, and make up to the mark with heptane. Shake thoroughly to mix. Allow the solution to stand for 10 min, and filter (see 6.3), if necessary, to remove insoluble material.

10.2.1.1 For samples in which the concentration of one or more aromatic hydrocarbon types falls outside the calibration range, prepare a more concentrated (for example, 2 g/10 mL) or more dilute (0.5 g/10 mL) sample solution as appropriate.

NOTE 24—If another dilution factor than the one suggested is used, it could modify the retention time and the amount calculated.

10.2.2 When operating conditions are steady (see 9.4) and identical to those used for obtaining the calibration data (see 10.1), inject 10 µL of the sample solution (see 10.2.1) and start data collection. Actuate the backflush valve at the predetermined time (see 9.6) to elute the T+AHs as a single sharp peak (see Fig. 4). When the analysis is finished, reverse the flow direction of the mobile phase (that is, return to forward flush) and allow the baseline to stabilize before injecting the next sample.

10.2.3 With reference to Fig. 5, devise a suitable method to find and identify correctly the MAHs, DAHs, and T+AHs. Fig. 5 shows a typical chromatogram for a sample of diesel fuel.

10.2.4 Draw a baseline from just before the beginning of the non-aromatics peak (*A* in Fig. 5) to a point on the chromatogram immediately before the backflush point (*D* in Fig. 5).

10.2.5 Drop a vertical line from the valley (*B* in Fig. 5) between non-aromatics and MAHs to the baseline.

10.2.6 Drop a vertical line from the valley (*C* in Fig. 5) between MAHs and DAHs to the baseline.

10.2.7 Draw a baseline from just before the T+AH peak (*E* in Fig. 5) to a point just after the T+AH compounds elute (*F* in Fig. 5). As some baseline disturbance is to be expected following actuation of the backflush valve, wait for the baseline to stabilize before drawing the baseline after the backflush point.

10.2.8 Integrate the area due to MAHs from points *B* to *C* (see Fig. 5).

10.2.9 Integrate the area due to DAHs from points *C* to *D* (see Fig. 5).

10.2.10 Integrate the area due to T+AHs from points *E* to *F* (see Fig. 5).

NOTE 25—If the chromatographic data have been processed automatically, visually check to see that the integration parameters have correctly identified and integrated the peaks.