



Designation: **D7807—12 D7807 – 20**

Standard Test Method for Determination of Boiling Range Distribution of Hydrocarbon and Sulfur Components of Petroleum Distillates by Gas Chromatography and Chemiluminescence Detection¹

This standard is issued under the fixed designation D7807; 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-~~Scope~~*

1.1 This test method covers the determination of the boiling range distribution of petroleum products. The test method is applicable to petroleum products and fractions having a final boiling point of ~~538°C (1000°F)~~ 538 °C (1000 °F) or lower at atmospheric pressure as measured by this test method. This test method is limited to samples having a boiling range greater than ~~55°C (100°F)~~ 55 °C (100 °F), and having a vapor pressure sufficiently low to permit sampling at ambient temperature.

1.1.1 The applicable sulfur concentration range will vary to some extent depending on the boiling point distribution of the sample and the instrumentation used; however, in most cases, the test method is applicable to samples containing levels of sulfur above ~~10–10 mg mg/kg/kg~~.

1.2 This test method requires the use of both FID and SCD for detection. The hydrocarbon simulated distillation data obtained from the FID signal should be performed according to Test Method ~~D2887~~; Procedure B.

1.3 The test method is not applicable for analysis of petroleum distillates containing low molecular weight components (for example, naphthas, reformates, gasolines, crude oils). Materials containing heterogeneous components (for example, alcohols, ethers, acids, or esters) or residue are not to be analyzed by this test method. See Test Methods ~~D3710, D7096, D5307, D7169, or D7500~~.

1.4 This test method does not purport to identify all sulfur species in a sample. The detector response to sulfur is equimolar for all sulfur compounds within the scope (1.1) of this test method. Thus, unidentified sulfur compounds are determined with equal precision to that of identified substances. Total sulfur content is determined from the total area of the sulfur detector.

1.4.1 This test method uses the principles of simulated distillation methodology.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure](#)

[D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure](#)

[D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry](#)

[D2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography](#)

[D2892 Test Method for Distillation of Crude Petroleum \(15-Theoretical Plate Column\)](#)

[D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry](#)

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 D02.04.0H on Chromatographic Distribution Methods.

Current edition approved Dec. 15, 2012; May 1, 2020. Published April 2013; June 2020. Originally approved in 2019. Last previous edition approved in 2019 as D7807 – 19. DOI: ~~10.1520/D7807-12~~ 10.1520/D7807-20.

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

- [D3710 Test Method for Boiling Range Distribution of Gasoline and Gasoline Fractions by Gas Chromatography \(Withdrawn 2014\)](#)³
- ~~[D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards](#)~~
- [D4626 Practice for Calculation of Gas Chromatographic Response Factors](#)
- [D5307 Test Method for Determination of Boiling Range Distribution of Crude Petroleum by Gas Chromatography \(Withdrawn 2011\)](#)³
- [D5504 Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Chemiluminescence](#)
- [D5623 Test Method for Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection](#)
- [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, Liquid Fuels, and Lubricants](#)
- [D6352 Test Method for Boiling Range Distribution of Petroleum Distillates in Boiling Range from 174 °C to 700 °C by Gas Chromatography](#)
- [D7096 Test Method for Determination of the Boiling Range Distribution of Gasoline by Wide-Bore Capillary Gas Chromatography](#)
- [D7169 Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High Temperature Gas Chromatography](#)
- [D7500 Test Method for Determination of Boiling Range Distribution of Distillates and Lubricating Base Oils—in Boiling Range from 100 °C to 735 °C by Gas Chromatography](#)
- [E178 Practice for Dealing With Outlying Observations](#)
- [E355 Practice for Gas Chromatography Terms and Relationships](#)
- [E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography](#)
- [E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs](#)

3. Terminology

3.1 *Definitions*—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions of these can be found in Practices [E355](#), [E594](#), and [E1510](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *area slice, n*—the area, resulting from the integration of the chromatographic detector signal, within a specified retention time interval. Peak detection parameters are bypassed and the detector signal integral is recorded as area slices of consecutive, fixed duration time intervals.

3.2.2 *corrected area slice, n*—an area slice corrected for baseline offset, by subtraction of the exactly corresponding area slice in a previously recorded blank (non-sample) analysis.

3.2.3 *cumulative corrected area, n*—the accumulated sum of corrected area slices from the beginning of the analysis through a given retention time, ignoring any non-sample area (for example, solvent).

3.2.4 *final boiling point (FBP), n*—the temperature (corresponding to the retention time) at which a cumulative corrected area count equal to ~~99.5 %~~ 99.5 % of the total sample area under the chromatogram is obtained.

3.2.5 *initial boiling point (IBP), n*—the temperature (corresponding to the retention time) at which a cumulative corrected area equal to ~~0.5 %~~ 0.5 % of the total sample area under the chromatogram is obtained.

3.2.6 *response factor (RF), n*—the factor used in order to calculate the mg/kg ~~Sulfur~~sulfur recovery of the sample.

3.2.7 *slice rate, n*—the time interval used to integrate the continuous (analog) chromatographic detector response during an analysis. The slice rate is expressed in Hz (for example, integrations or slices per second).

3.2.8 *slice time, n*—the cumulative slice rate (analysis time) associated with each area slice throughout the chromatographic analysis. The slice rate is the time at the end of each contiguous area slice.

3.2.9 *total sample area, n*—the cumulative corrected area from the initial point to the final area point.

3.3 *Abbreviations*—A common abbreviation of hydrocarbon compounds is to designate the number of carbon atoms in the compound. A prefix is used to indicate the carbon chain form, while a subscripted suffix denotes the number of carbon atoms (for example, normal decane n-C10, iso-tetradecane = i-C14).

4. Summary of Test Method

4.1 The boiling range distribution determination by distillation is simulated by the use of gas chromatography. A nonpolar open tubular (capillary) gas chromatographic column is used to elute the hydrocarbon components of the sample in order of increasing

³ The last approved version of this historical standard is referenced on www.astm.org.

boiling point. The column temperature is raised at a reproducible linear rate and the area under the chromatogram is recorded throughout the analysis. Boiling points are assigned to the time axis from a calibration curve obtained under the same chromatographic conditions by analyzing a known mixture of hydrocarbons covering the boiling range expected in the sample. A quantitative standard is used to determine the SCD detector response factor. Finally, the sample solution is injected and with the use of the response factor, the amount of sample recovered is calculated. After converting the retention times of the sample slices to temperature, the boiling point distribution can be calculated up to the recovered amount. From these data, the boiling range distribution can be obtained.

4.1.1 By splitting the column effluent to FID and Sulfur Chemiluminescence Detector, simultaneous detection for Hydrocarbonhydrocarbon (FID) and Sulfursulfur (SCD) components boiling range distribution is obtained. The Hydrocarbonhydrocarbon simulated distillation data should be calculated according to Test Method **D2887**.

4.1.2 Alternatively, the FID may be used with the SCD detector superimposed over the FID and thus avoiding splitting the sample through the column exit. This type of arrangement will lower the sensitivity of the detector in the sulfur mode.

4.2 A sample aliquot is introduced into the chromatographic system. Sample vaporization is provided by separate heating of the point of injection or in conjunction with column oven heating.

4.3 The column oven temperature is raised at a reproducible linear rate to effect separation of the sample components in order of increasing boiling point. The elution of sample components is quantitatively determined using a flame ionization detector and a sulfur chemiluminescence detector. The detector signal integral is recorded as area slices for consecutive retention time intervals during the analysis.

4.4 Retention times of known normal paraffin hydrocarbons spanning the scope of this test method (C5- C44) are determined and correlated to their theoretical boiling point temperatures. The normalized cumulative corrected sample areas for each consecutive recorded time interval are used to calculate the boiling range distribution. The boiling point temperature at each reported percent off increment is calculated from the retention time calibration.

4.5 *Sulfur Chemiluminescence Detection*—As sulfur compounds elute from the gas chromatographic column, they are processed in a heated combustion zone. The products are collected and transferred to a sulfur chemiluminescence detector (SCD). This technique provides a sensitive, selective, linear response to volatile sulfur compounds and is used for the selective sulfur detection, while collecting hydrocarbon data from the FID.

4.6 *Alternative Detectors*—This test method is written for the sulfur chemiluminescence detector but other sulfur specific detectors can be used provided they have sufficient linearity, sensitivity, and have equimolar response to all eluted sulfur compounds, do not suffer from interferences, and satisfy quality assurance criteria. Regulatory agencies may require demonstration of equivalency of alternative detection systems to the SCD.

5. Significance and Use

ASTM D7807-20

5.1 The boiling range distribution of light and medium petroleum distillate fractions provides an insight into the composition of feed stocks and products related to petroleum refining processes. This gas chromatographic determination of boiling range can be used to replace conventional distillation methods for control of refining operations. This test method can be used for product specification testing with the mutual agreement of interested parties.

5.2 This test method extends the scope of Test Method **D2887** (538°C)(538 °C) boiling range determination by gas chromatography to include sulfur boiling range distribution in the petroleum distillate fractions. Knowledge of the amount of sulfur and its distribution in hydrocarbons is economically important in determining product value and in determining how best to process or refine intermediate products. Sulfur compounds are known to affect numerous properties of petroleum and petrochemical products. The corrosion of metals and poisoning of catalysts is of particular concern. In addition, the content of sulfur in various refined products may be subject to governmental regulations. Test Methods, such as, **D2622**, **D3120**, **D5504** and **D5623**, are available to determine total sulfur content or content of individual sulfur compounds in petroleum and petroleum products. Test Methods, such as, **D86**, **D1160**, **D2887**, **D3710**, and **D2892**, are also available to determine the hydrocarbon boiling ranges of such samples. The gas chromatographic determination of the sulfur boiling range assists in process development, in treatment and control of refining operations and is useful for assessing product quality. This determination produces detailed information about the sulfur distribution in a sample that cannot be obtained by either total sulfur analysis or analysis of sulfur in discreet distillation cuts.

5.2.1 The hydrocarbon boiling range distributions obtained by Test Method **D2887** are theoretically equivalent to those obtained by true boiling point (TBP) distillation (see Test Method **D2892**). They are not equivalent to results from low efficiency distillation such as those obtained with Test Method **D86** or **D1160**.

6. Apparatus

6.1 *Chromatograph*—Any gas chromatograph, with hardware necessary for interfacing to a chemiluminescence detector and containing all features necessary for the intended application(s) can be used. The gas chromatographic system used shall have the following performance characteristics:

6.2 *Column Temperature Programmer*—The chromatograph must be capable of linear programmed temperature operation over a range sufficient to elute compounds up to a boiling temperature of ~~538°C (1000°F)~~ 538 °C (1000 °F) before reaching the upper end of the temperature program. The programming rate must be sufficiently reproducible to obtain retention time repeatability of ~~0.01 min (0.6 s), 0.01 min (0.6 s,~~ corresponding to approximately ~~0.5°C)~~ 0.5 °C for each component in the calibration mixture described in 7.7.

6.3 *Detectors*—This test method requires a Flame Ionization Detector (FID) and a Sulfur Chemiluminescence Detector (SCD).

6.3.1 *FID*—The FID shall meet or exceed the following specifications in accordance with Practice E594. Check the detector according to the instrument manufacturer's instructions.

6.3.2 *SCD*—The sulfur chemiluminescence detector shall meet or exceed the following specifications: (1) greater than 10^3 linearity, (2) less than ~~1 pg~~ 1 pg S/s sensitivity, (3) greater than 10^6 selectivity for sulfur compounds over hydrocarbons, (4) no quenching of sulfur compound response from co-eluting hydrocarbons when the same volume of sample is injected as for regular analysis, and (5) equimolar response (~~$\leq \pm 10\%$~~) ($\leq \pm 10\%$) on a sulfur basis.

6.3.2.1 For the purpose of boiling point calibration, the system must be capable of measuring sulfur compounds and hydrocarbons simultaneously from a single column and injection, for example, flame ionization detector with splitting the column effluent prior to the sulfur chemiluminescence detector. Alternatively, a combined FID/SCD can also be used in order to obtain simultaneous sulfur and FID chromatogram.

6.3.2.2 Sulfur compounds eluting from the chromatographic column are processed in a heated ~~hydrogen-rich~~ hydrogen-rich combustion zone fitted to the end of the column. Products are transferred under reduced pressure to the reaction chamber of the chemiluminescence detector. An excess of ozone present in the chamber reacts with the sulfur combustion product(s) to liberate blue ~~(480 nm)~~ (480 nm) and ultraviolet light ~~(260 nm)~~ (260 nm).

6.3.3 *Detector Split Requirements*—To ensure the low levels of sulfur are detected properly, the system must be capable to detect the components in the system sulfur test mixture (see 8.9) with signal to noise (peak-to-peak) ratio of at least 100. Connections of the column to the detector shall be such that no temperature below the column temperature exists. Refer to Practice E1510 for proper installation and conditioning of the capillary column.

6.4 *Sample Inlet System*—Any sample inlet system capable of meeting the performance specification in 8.7 may be used. Programmed temperature vaporization (PTV) and programmable cool on-column injection systems have been used successfully.

6.5 *Carrier Gas Flow Control*—The chromatograph shall be equipped with carrier gas pressure or flow control capable of maintaining constant carrier gas flow control through the column throughout the column temperature program cycle.

6.6 *Micro syringe*—*Syringe*—A micro syringe with a 23 gauge or smaller stainless steel needle is used for sample introduction. Syringes of ~~0.1 to 10~~ 0.1 to 10 μL capacity are commercially available. Automatic syringe injection is recommended.

6.7 *Column*—This test method is limited to the use of non-polar wall coated open tubular (WCOT) columns.

6.7.1 Any column and conditions may be used that provide separation of typical petroleum hydrocarbons in order of increasing boiling point and meet the column performance requirements of 8.7.1 and 9.3.1.1.

6.7.2 Glass, fused silica, and stainless steel columns, with a ~~0.53 mm~~ 0.53 mm diameter have been successfully used. Cross-linked and bonded ~~100%~~ 100% dimethyl-polysiloxane stationary phases with film thickness of ~~0.50.5~~ 0.50.5 μm to ~~2.65~~ 2.65 μm have been used. The column length and liquid phase film thickness shall allow the elution of at least C44 n-paraffin (BP = ~~545°C~~) 545 °C).

6.8 *Data Acquisition System*—Use of an electronic integrating device or computer is mandatory for determining the detector response and for boiling point calibration. The device must have the following capabilities: (1) graphic presentation of the chromatogram, (2) digital display of chromatographic peak areas, (3) measurement of area and time intervals, (4) calculation and use of response factors in accordance with Practice D4626, for example, external standardization, and (5) the maximum area measured must be within the linear range of the measuring system used.

NOTE 1—Some gas chromatographs have an algorithm built into their operating software that allows a mathematical model of the baseline profile to be stored in memory. This profile is automatically subtracted from the detector signal on subsequent sample runs to compensate for the column bleed. Some integration systems also store and automatically subtract a blank analysis from subsequent analytical determinations.

7. Reagents and Materials

7.1 *Carrier Gas*—Helium, of at least ~~99.999%~~ 99.999% (v/v) purity (**Warning**—Helium is a compressed gas under high pressure). Any oxygen present is removed by a chemical resin filter (**Warning**—Follow the safety instructions from the filter supplier.) The total amount of impurities should not exceed ~~10 mL/m³~~ 10 mL/m³. Helium or Nitrogen (at least ~~99.999%~~ 99.999% (v/v)) can also be used as detector make-up gas.

7.1.1 Additional purification is recommended by the use of molecular sieves or other suitable agents to remove water, oxygen, and hydrocarbons. Available pressure shall be sufficient to ensure a constant carrier gas flow rate.

7.2 *Hydrogen*—Hydrogen of at least ~~99.999%~~ 99.999% (v/v) purity (suitable for the flame ionization detector (FID)). (**Warning**—Hydrogen is an extremely flammable gas under high pressure.)

TABLE 1 Typical Gas Chromatographic Conditions

Instrument	A gas chromatograph equipped with an on-column or temperature programmable vaporizing injector (PTV)
Column	Capillary 10 m, 0.53 mm ID, 0.88 μm 100 % dimethyl-polysiloxane stationary phase
Flow Conditions	25 mL/min He carrier (constant flow)
Inlet <u>Inlet</u>	Temperature: Programmed 100°C to 350°C at 25°C/min <u>Temperature: Programmed 100 °C to 350 °C at 25 °C/min</u>
Detector	Split FID – SCD or SCD with FID Adapter
FID <u>FID</u>	Temperature: 350°C <u>Temperature: 350 °C</u> 35 mL/min H ₂ , 350 mL/min Air
SCD <u>SCD</u>	Temperature: 950°C <u>Temperature: 950 °C</u> 10 mL/min O ₂ , 90 mL/min H ₂ 100 – 150 mL/min He or N ₂ make-up 100 mL/min to 150 mL/min He or N ₂ make-up
Oven <u>Oven</u>	35°C to 350°C at 25°C/min <u>35 °C to 350 °C at 25 °C/min</u>
Sample Injection	0.1 μL neat

7.3 *Air*—High purity (for example, hydrocarbon-free) compressed air is used as the oxidant for the flame ionization detector (FID). (**Warning**—Compressed air is a gas under high pressure and supports combustion.)

7.4 *Oxygen*—High purity (for example, hydrocarbon-free) compressed oxygen is used as the oxidant for the sulfur chemiluminescence detector (SCD). (**Warning**—~~compressed~~ Compressed oxygen is a gas under high pressure and supports combustion.)

NOTE 2—Some SCD detectors allow the use of air instead of oxygen; oxygen; contact the SCD manufacturer for information on the use of air as oxidant.

7.5 *Solvents*—~~unless~~ Unless otherwise indicated, it is intended that all solvents conform to the specifications of the committee on analytical Reagents of the American Chemical Society⁴ where such specifications are available.

7.5.1 Other grades may be used provided it is first ascertained that the solvent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.6 *Cyclohexane* (C_6H_{12})—, (99+ % (99+ % pure) may be used as a viscosity reducing solvent. It is miscible with asphaltic hydrocarbons, however, it responds well to the FID. The quality (hydrocarbon content) should be determined by this test method prior to use as a sample diluent. (**Warning**—Cyclohexane is flammable.)

7.7 *Calibration Mixture*—An accurately weighed mixture of approximately equal mass quantities of ~~nhydrocarbons~~ hydrocarbons dissolved in a suitable solvent. The mixture shall cover the boiling range from n-C5 to n-C44, but does not need to include every carbon number, but at least sufficient number of points to generate a reliable calibration curve and the C16 and C18.

7.7.1 At least one compound in the mixture must have a boiling point lower than the IBP of the sample and at least one compound in the mixture must have a boiling point higher than the FBP of the sample. Boiling points of ~~n-paraffin~~ n-paraffins are listed in **Table 2**.

7.7.2 If necessary, for the calibration mixture to have a compound with a boiling point below the IBP of the sample, propane or butane can be added to the calibration mixture, non-quantitatively, by bubbling the gaseous compound into the calibration mixture in a septum sealed vial using a gas syringe.

7.7.3 *Reference Material*—A reference sample that has been analyzed by laboratories participating in the test method cooperative study. Consensus values for the boiling range distribution of this sample are being determined.

8. Preparation of Apparatus

8.1 Gas Chromatograph Setup:

8.2 Place the gas chromatograph and ancillary equipment into operation in accordance with the manufacturer's instructions. Recommended operating conditions are shown in **Table 1**.

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

TABLE 2 Boiling Points of Normal Paraffins^{A,B}

Carbon No.	Boiling Point, °C	Boiling Point, °F	Carbon No.	Boiling Point, °C	Boiling Point, °F
1	-162	-259	23	380	716
2	-89	-127	24	391	736
3	-42	-44	25	402	755
4	0	31	26	412	774
5	36	97	27	422	791
6	69	156	28	431	808
7	98	209	29	440	825
8	126	258	30	449	840
9	151	303	31	458	856
10	174	345	32	466	870
11	196	385	33	474	885
12	216	421	34	481	898
13	235	456	35	489	912
14	254	488	36	496	925
15	271	519	37	503	937
16	287	548	38	509	948
17	302	576	39	516	961
18	316	601	40	522	972
19	330	626	41	528	982
20	344	651	42	534	993
21	356	674	43	540	1004
22	369	695	44	545	1013

^A API Project 44, October 31, 1972 is believed to have provided the original normal paraffin boiling point data that are listed in Table 1. However, over the years some of the data contained in both API Project 44 (Thermodynamics Research Center Hydrocarbon Project) and Test Method D6352 have changed and they are no longer equivalent. Table 1 represents the current normal paraffin boiling point values accepted by Subcommittee D02.04 and found in all test methods under the jurisdiction of Section D02.04.0H.

^B Used *n*-paraffin boiling points are traditionally rounded to the nearest whole degree for calibration. The boiling points listed in Table 1 are correct to the nearest whole number in both degrees Celsius and degrees Fahrenheit. However, if a conversion is made from one unit to the other and then rounded to a whole number, the results will not agree with the table values for a few carbon numbers. For example, the boiling point of *n*-heptane is 98.425°C, 98.425 °C, which is correctly rounded to 98°C, 98 °C in the table. However, converting 98.425°C to 209.165°F, 209.165 °F, which rounds to 209°F, 209 °F, while converting 98°C to 208.4°F, 208.4 °F, which rounds to 208°F, 208 °F. Carbon numbers 2, 4, 7, 8, 9, 13, 14, 15, 16, 25, 27, and 32 are affected by rounding.

8.3 When attaching the column to the detector inlet, ensure that the end of the column terminates as close as possible to the FID jet. Follow the instructions in Practice E1510.

8.4 The FID should be periodically inspected and, if necessary, remove any foreign deposits formed in the detector from combustion of silicone liquid phase or other materials. Such deposits will change the response characteristics of the detector.

8.5 SCD—Place in service in accordance with the manufacturer’s instructions. Optimization of the oxidant/fuel ratio is critical for ensuring complete combustion of hydrocarbon components in a sample. A flame or combustion zone that is too hydrogen rich will result in incomplete combustion. Matrix interference is occasionally observed when changing sample size. When matrix interference is indicated, samples may be analyzed by dilution or application of other mitigation efforts.

8.5.1 The typical flameless/combustion zone interface contains ceramic tubes in its construction. The performance of these tubes is critical to performance of the SCD system. Compromised ceramic tubes are susceptible to matrix effects. Compromised tubes may allow for reproducible duplicate sample analysis but will fail QA procedures such as matrix dilution and spike analyses. Poorly functioning tubes can also result in severe instrument drift, loss of equimolar response, and general response instability. Compromised tubes must be replaced to restore nominal instrument function.

8.6 Column Conditioning—A new column will require conditioning at the upper test method operating temperature to reduce or eliminate significant liquid phase bleed, resulting in a stable chromatographic baseline. Follow the guidelines outlined in Practice E1510.

8.7 System Performance Specification:

8.7.1 Column Resolution—The column resolution, influenced by both the column physical parameters and operating conditions, affects the overall determination of boiling range distribution. Resolution is therefore specified to maintain equivalence between different systems (laboratories) employing this test method. Resolution is determined using Eq 1 and the C16 and C18 paraffins from the calibration mixture analysis (see 7.7), and is illustrated in Fig. 1. Resolution (R) should be at least three, using the identical conditions employed for sample analyses:

$$R = 2(t_2 - t_1) / (1.699(w_2 + w_1)) \quad (1)$$

where:

- R = resolution,
- t₇ = time(s) for the *n*-C16 peak maximum,
- t₁ = time(s) for the *n*-C16 peak maximum,
- t₂ = time(s) for the *n*-C18 peak maximum,

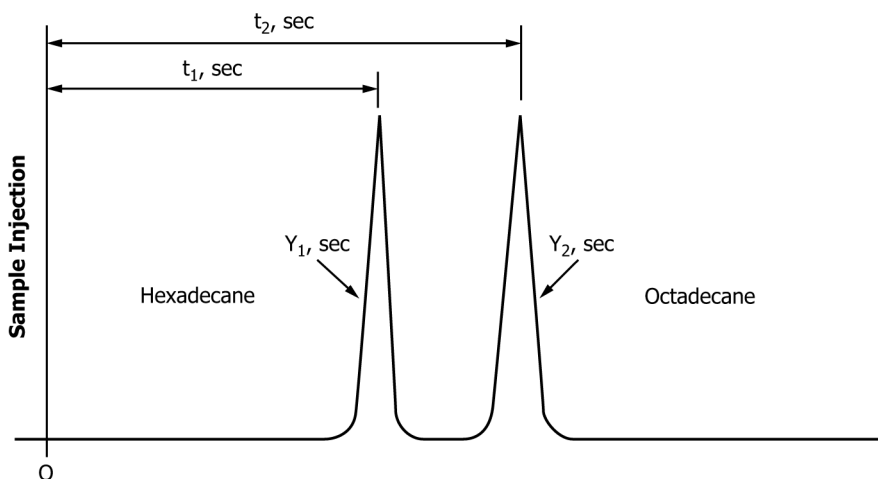


FIG. 1 Column Resolution Parameters

w_1 = peak width(s), at half height, of the *n*-C16 peak, and
 w_2 = peak width(s), at half height, of the *n*-C18 peak.

8.7.2 *Column Elution Characteristics*—The recommended column liquid phase is a non-polar phase such as ~~100 %~~ 100 % dimethyl-polysiloxane.

8.8 *Sulfur Compound Standards*—~~99 + %~~ 99 + % purity (if available). Obtain pure standard material of all sulfur compounds of interest (**Warning**—Sulfur compounds can be flammable and harmful or fatal when ingested or inhaled.). If purity is unknown or questionable, analyze the individual standard material by any appropriate means and use the result to provide accurate standard quantities.

8.9 *System Sulfur Test Mixture*—Gravimetrically prepare a solution of sulfur compounds at approximately 20 mg/kg sulfur in ultra-low sulfur Diesel in accordance with Practice D4307. The test mixture should cover a broad boiling point range and should be within the range of the hydrocarbon distribution.

8.9.1 For example, Benzothiophene, DiBenzothiophene, and 2,2'-dithiopyridine:

Name	Formula	Mol wt	m/m%, S	Boiling Point, °C
Benzothiophene	C ₈ H ₆ S	134.2	23.89 %	221
Dibenzothiophene	C ₁₂ H ₈ S	184.26	17.40 %	332
2,2'-dithiopyridine	C ₁₀ H ₈ N ₂ S ₂	220.31	29.10 %	348

8.8 *Sulfur Equimolarity—Standard*—The SCD is an equimolar detector. Therefore, response factors for all sulfur components should be within 10 %. Failure to satisfy this criterion indicates either system test mixture degradation or failure of the SCD heated A petroleum sample with a known total Sulfur content and a known boiling point distribution of the sulfur (**Warning**—combustion zone or in other parts of the analysis system. Sulfur compounds can be flammable and harmful or fatal when ingested or inhaled.).

8.8.1 Calculate the relative response factor for each sulfur compound in the System Sulfur test mixture the Sulfur Standard (see 8-98.8);).

8.8.2 Inject and analyze a suitable amount of the system-sulfur test mixture standard (8-98.8). Relative response factors should shall be calculated for each sulfur compound in the test mixture (relative to a referenced component) the Sulfur standard in accordance with Practice D4626 or Eq 2.

$$R_m = \frac{C_n \times A_r}{C_r \times A_n} \quad (2)$$

where:

- R_m = relative response factor for a given sulfur compound,
- R_m = relative response factor for the Sulfur standard,
- C_n = concentration of the sulfur compound as sulfur,
- A_n = peak area of sulfur compound,
- C_r = concentration of referenced sulfur standard as sulfur, and
- A_r = peak area of the referenced sulfur standard.

8.10.2.1 The relative response factor (R_m) for each sulfur compound should not deviate from the average response factor by more than 10 %. Deviation of response by more than 10 % or severe peak asymmetry indicates a chromatography or detector