Designation: D7213 – 15

Standard Test Method for Boiling Range Distribution of Petroleum Distillates in the Boiling Range from 100 °C to 615 °C by Gas Chromatography[1]

This standard is issued under the fixed designation D7213; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of the boiling range distribution of petroleum products. This test method is applicable to petroleum distillates having an initial boiling point greater than 100 °C and a final boiling point less than 615 °C at atmospheric pressure as measured by this test method.

1.2 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 D7096, D2887, D6352, or D7169.

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

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure

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)

D4626 Practice for Calculation of Gas Chromatographic Response Factors

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

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. In area slice mode (see 6.4.2), 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.

[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.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

3.2.3 *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 % 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 count equal to 0.5 % of the total sample area under the chromatogram is obtained.

3.2.6 *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).

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

3.2.8 *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-C_{10}; iso-tetradecane = i-C_{14}).

4. Summary of Test Method

4.1 The boiling range distribution by distillation is simulated by the use of gas chromatography. The solvent should not interfere with measurement of the sample in the 100 °C to 615 °C range, and it should be apolar. A non-polar open tubular (capillary) gas chromatographic column is used to elute the hydrocarbon components of the sample in order of increasing boiling point.

4.2 A sample aliquot is diluted with a viscosity reducing solvent and 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 hydrocarbon components in order of increasing boiling point. The elution of sample components is quantitatively determined using a flame ionization 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 (C_5-C_{60}) are determined and correlated to their 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.

5. Significance and Use

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 process, 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 boiling range determination by gas chromatography to include light and medium petroleum distillate fractions beyond the scope of Test Method D2887 (538 °C) and below Test Method D6352 (700 °C).

5.3 Boiling range distributions obtained by this test method 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*—The gas chromatographic system used shall have the following performance characteristics:

6.1.1 *Column Oven*—Capable of sustained and linear programmed temperature operation from near ambient (for example, 35 °C to 50 °C) up to 400 °C.

6.1.2 *Column Temperature Programmer*—The chromatograph shall be capable of linear programmed temperature operation up to 400 °C at selectable linear rates up to 20 °C/min. The programming rate shall be sufficiently reproducible to obtain the retention time repeatability of 0.1 min (6 s) for each component in the calibration mixture described in 7.5.

6.1.3 *Detector*—This test method requires a flame ionization detector (FID). The detector shall meet or exceed the following specifications as detailed in Practice E594. The flame jet should have an orifice of approximately 0.45 mm to 0.50 mm.

6.1.3.1 *Operating Temperature,* 400 °C.

6.1.3.2 *Sensitivity,* >0.005 coulombs/g carbon.

6.1.3.3 *Minimum Detectability,* 1×10^{-11} g carbon/s.

6.1.3.4 *Linear Range,* $>10^6$.

6.1.3.5 Connection 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.

6.1.4 *Sample Inlet System*—Any sample inlet system capable of meeting the performance specification in 7.6 may be used. Programmed temperature vaporization (PTV) and programmable cool on-column injection systems have been used successfully.

6.1.5 *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.

6.2 *Microsyringe*—A microsyringe with a 23 gauge or smaller stainless steel needle is used for on-column sample introduction. Syringes of 0.1 µL to 10 µL capacity are available.

6.2.1 Automatic syringe injection is recommended to achieve best precision.

6.3 *Column*—This test method is limited to the use of non-polar wall coated open tubular (WCOT) columns of high thermal stability. Glass, fused silica, and stainless steel columns, with a 0.53 mm diameter have been successfully used. Cross-linked or bonded 100 % dimethyl-polysiloxane stationary phases with film thickness of 0.5 µm to 1.0 µm have been used. The column length and liquid phase film thickness shall allow the elution of at least C_{60} *n*-paraffin (BP = 615 °C). The column and conditions shall provide separation of typical petroleum hydrocarbons in order of increasing boiling point and meet the column resolution requirements of 8.2.1. The column shall provide a resolution between one and ten using this test method's operating conditions.

6.4 *Data Acquisition System:*

6.4.1 *Recorder*—A 0 mV to 1 mV range recording potentiometer or equivalent, with a full-scale response time of 2 s or less may be used to provide a graphical display.

6.4.2 *Integrator*—Means shall be provided for determining the accumulated area under the chromatogram. This can be done by means of an electronic integrator or computer-based chromatography data system. The integrator/computer system shall have normal chromatographic software for measuring the retention time and areas of eluting peaks (peak detection mode). In addition, the system shall be capable of converting the continuously integrated detector signal into area slices of fixed duration (area slice mode). These contiguous area slices, collected for the entire analysis, are stored for later processing. The electronic range of the integrator/computer (for example, 1 V, 10 V) shall be operated within the linear range of the detector/electrometer 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[3]

7.1 *Carrier Gas*—Helium or hydrogen of high purity. (**Warning—**Helium and hydrogen are compressed gases under high pressure; hydrogen is an extremely flammable gas under high pressure.) These gases may be used as the carrier gas and should not contain more than 5 mL/m^3 of oxygen. The total amount of impurities should not exceed 10 mL/m^3. 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 high purity (for example, hydrocarbon-free) is used as fuel for the flame ionization detector (FID). (**Warning—**Hydrogen is an extremely flammable gas under high pressure.)

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 *Solvents*—Unless otherwise indicated, it is intended that all solvents conform to the specifications of the committee on analytical Reagents of the American Chemical Society where such specifications are available.[3] 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.

7.4.1 *Carbon Disulfide (CS_2)*—(99+ % pure) may be used as a viscosity reducing solvent and as a means of reducing mass of sample introduced onto the column to ensure linear detector response and reduced peak skewness. It is miscible with asphaltic hydrocarbons and provides a relatively small response with the FID. The quality (hydrocarbon content) should be determined by this test method prior to use as a sample diluent. (**Warning—** Carbon disulfide is extremely flammable and toxic.)

7.5 *Cyclohexane (C_6H_{12})*—(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.)

7.6 *Calibration Mixture*—A qualitative mixture of *n*-paraffins (nominally C_5 to C_{60}) dissolved in a suitable solvent. The final concentration should be approximately one part of *n*-paraffin mixture to one hundred parts of solvent. At least one compound in the mixture shall have a boiling point lower than the initial boiling point of the sample being analyzed, as defined in the scope of this test method (1.1). The calibration mixture shall contain at least 13 known *n*-paraffins (for example, C_6, C_7, C_8, C_9, C_{10}, C_{12}, C_{16}, C_{20}, C_{30}, C_{40}, C_{50}, C_{52}, C_{60}). Boiling points of *n*-paraffins are listed in Table 1.

NOTE 2—A suitable calibration mixture can be obtained by dissolving a polyolefin wax (for example, Polywax 500[4]) in a volatile solvent (for example, carbon disulfide or cyclohexane). Solutions of one part Polywax to one hundred parts solvent can be prepared. Lower boiling point paraffins will have to be added to insure conformance with 7.6. Fig. 1 illustrates a typical calibration mixture chromatogram.

7.7 *Response Linearity Mixture*—Prepare a quantitatively weighed mixture of at least ten individual paraffins (>99 % purity), covering the boiling range of the test method. The highest boiling point component should be at least n-C_{60} . The mixture shall contain n-C_{40}. Use a suitable solvent to provide

[3] *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 *Annual 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.

[4] Polywax is a trademark of the Baker Petrolite Corporation, Barnsdall, OK.

TABLE 1 Boiling Points of *n*-Paraffins[A,B]

Carbon Number	Boiling Point °C	Boiling Point °F
5	36	97
6	69	156
7	98	209
8	126	258
9	151	303
10	174	345
11	196	385
12	216	421
13	235	456
14	254	488
15	271	519
16	287	548
17	302	576
18	316	601
19	330	626
20	344	651
21	356	674
22	369	695
23	380	716
24	391	736
25	402	755
26	412	774
27	422	791
28	431	808
29	440	825
30	449	840
31	458	856
32	466	870
33	474	885
34	481	898
35	489	912
36	496	925
37	503	937
38	509	948
39	516	961
40	522	972
41	528	982
42	534	993
43	540	1004
44	545	1013
45	550	1022
46	556	1033
47	561	1042
48	566	1051
49	570	1058
50	575	1067
51	579	1074
52	584	1083
53	588	1090
54	592	1098
55	596	1105
56	600	1112
57	604	1119
58	608	1126
59	612	1134
60	615	1139

[A] API Project 44, 72-10-31, 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 D7213 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.H.
[B] Test Method D7213 has traditionally used *n*-paraffin boiling points 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 which is correctly rounded to 98 °C in the table. However, converting 98.425 °C gives 209.165 °F, which rounds to 208 °F, while converting 98 °C gives 208.4 °F, which rounds to 208 °F. Carbon numbers 2, 4, 7, 8, 9, 13, 14, 15, 16, 25, 27, and 32 are affected by rounding.

a solution of each component at approximately 0.5 % to 2.0 % by mass.

7.8 *Reference Material*—A reference sample that has been analyzed by laboratories participating in the test method

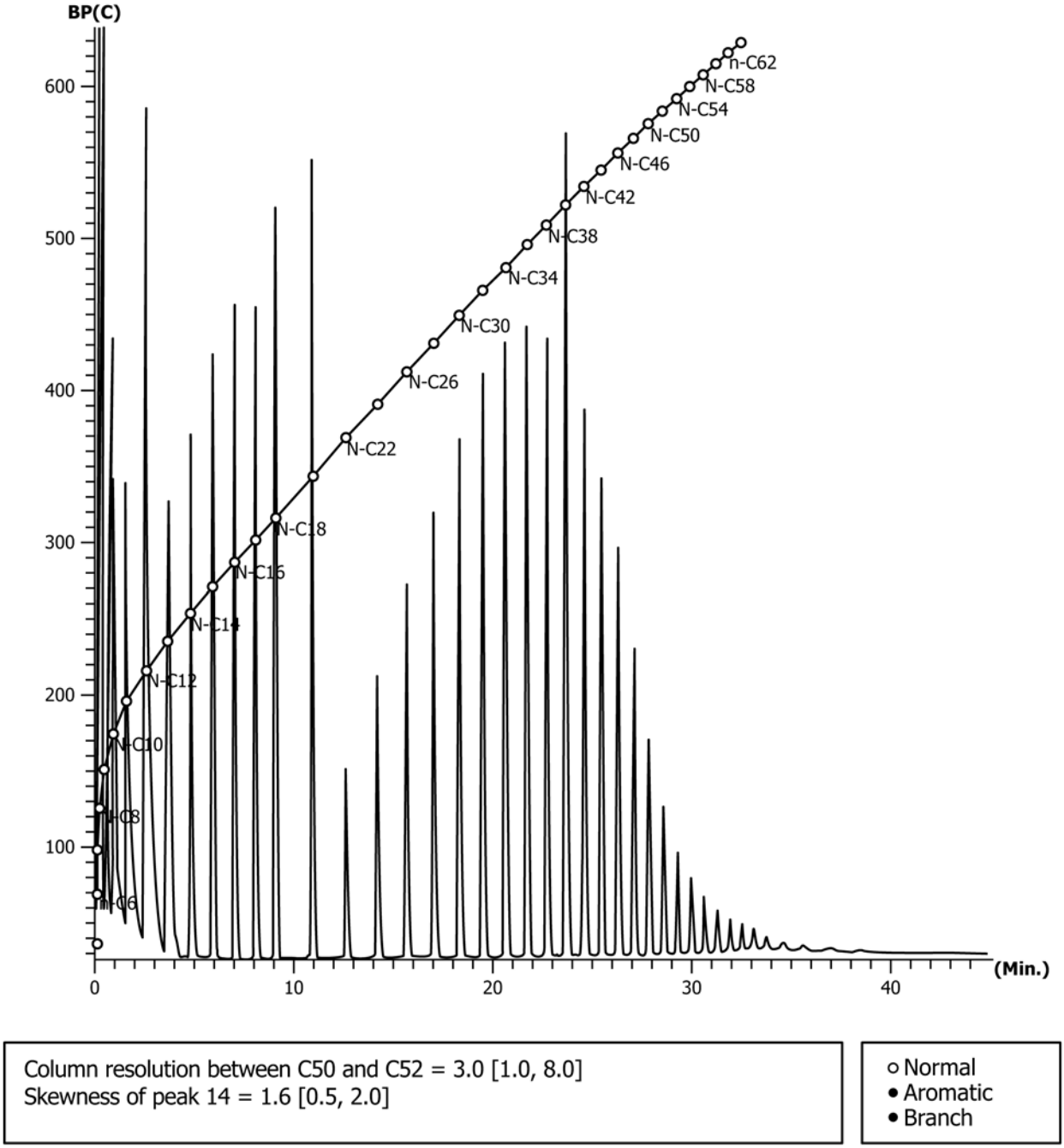

FIG. 1 Typical Calibration Curve with Plot

cooperative study. Consensus values for the boiling range distribution of this sample are being determined.

8. Preparation of Apparatus

8.1 *Gas Chromatograph Setup:*

8.1.1 Place the gas chromatograph and ancillary equipment into operation in accordance with the manufacturers instructions. Recommended operating conditions are shown in Table 2.

8.1.2 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.

8.1.3 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.

TABLE 2 Recommended Operating Conditions[A]

Injector	Cool on-column. Temperature Programmable Inlet (no Split)
Injection temperature	Oven-track mode
Auto sampler	Required for best precision
Data collection	Data is collected as independent area slices (average data collection rate is 1.0 Hz or 3.3 slices per second)
Column	Capillary, 5 m by 0.53 mm id film thickness; 0.1 µm to 1.0 µm (polymethylsiloxane) 0.8 µm – 1.0 µm was used in the ILS study
Flow conditions	UHP helium at 12 mL/min (constant flow)
Carrier: He and Hydrogen were used in the ILS Study	(make-up gas helium at 18 mL/min)
Detector	Flame Ionization; Temperature: 390 °C
Oven program	Initial oven temperature 35 °C – 50 °C, initial hold 0 min, program rate 10 °C/min., final oven temperature 380 °C, final hold 12 min, equilibration time 2 min
Sample size	1 µL
Sample dilution	2 mass percent in carbon disulfide
Calibration dilution	1 mass percent in carbon disulfide

[A] Hydrogen was used in the precision study. Results were found to be "statistically equivalent." See Research Report RR:D02-1725.

8.1.4 The inlet liner and initial portion of the column shall be periodically inspected and replaced if necessary to remove extraneous deposits or sample residue.

8.1.5 *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.

8.2 *System Performance Specification:*

8.2.1 *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 C_{50} and C_{52} paraffins from a calibration mixture analysis (or a polywax retention time boiling point mixture) (see 7.6). Resolution (R) should be at least one and not more than ten, 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,
t_1 = time for the n-C_{50} peak maximum,
t_2 = time for the n-C_{52} peak maximum,
w_1 = peak width, at half height, of the n-C_{50} peak and,
w_2 = peak width, at half height, of the n-C_{52} peak.

8.2.2 *Detector Response Calibration*—This test method assumes that the FID response to petroleum hydrocarbons is proportional to the mass of individual components. This shall be verified when the system is put in service, and whenever any changes are made to the system or operational parameters. Analyze the response linearity mixture (see 7.7) using the identical procedure to be used for the analysis of samples (see Section 9). Calculate the relative response factor for each *n*-paraffin (relative to *n*-tetracontane) as per Practice D4626 and Eq 2:

$$F_n = (M_n/A_n)/(M_{40}/A_{40}) \quad (2)$$

where:

F_n = relative response factor,
M_n = mass of the *n*-paraffin in the mixture,
A_n = peak area of the *n*-paraffin in the mixture,
M_{40} = mass of the *n*-tetracontane in the mixture, and
A_{40} = peak area of the *n*-tetracontane in the mixture.

8.2.2.1 The relative response factor (F_n) of each *n*-paraffin shall not deviate from unity by more than ±5 %.

8.2.3 *Column Temperature*—The column temperature program profile is selected such that the C_5 peak can be differentiated from the solvent and that the maximum boiling point (615 °C) *n*-paraffin (C_{60}) is eluted from the column before reaching the end of the temperature program. The actual program rate used will be influenced by other operating variables such as column dimensions, liquid phase film thickness, carrier gas and flow rate, and sample size.

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

9. Procedure

9.1 *Analysis Sequence Protocol*—Define and use a predetermined schedule of analysis events designed to achieve maximum reproducibility for these determinations. The schedule will include cooling the column oven and injector to the initial starting temperature, equilibration time, sample injection and system start, analysis, and final temperature hold time.

9.1.1 After chromatographic conditions have been set to meet performance requirements, program the column temperature upward to the maximum temperature to be used and hold that temperature for the selected time. Following the analysis sequence protocol, cool the column to the initial starting temperature.

9.1.2 During the cool down and equilibration time, ready the integrator/computer system. If a retention time calibration

is being performed, use the peak detection mode. For samples and baseline compensation (with or without solvent injection), use the area slice mode operation. The recommended slice rate for this test method is 1.0 Hz (3.3 slices per second). Other slice rates may be used if within the limits of 0.02 % and 0.2 % of the retention time of the final calibration component (C_{60}). Faster slice rates may be used, as may be required for other reasons, if provision is made to accumulate (bunch) the slice data to within these limits prior to determination of the boiling range distribution.

9.1.3 At the exact time set by the schedule, inject either the calibration mixture, solvent, or sample into the chromatograph; or make no injection (baseline blank). At the time of injection, start the chromatograph time cycle and the integrator/computer data acquisition. Follow the analysis protocol for all subsequent repetitive analyses or calibrations. Since complete resolution of sample peaks is not expected, do not change the sensitivity setting during the analysis.

9.2 *Baseline Blank*—Perform a blank analysis (baseline blank) at least once per day. The blank analysis may be without injection or by injection of an equivalent solvent volume as used with sample injections, depending upon the subsequent data handling capabilities for baseline/solvent compensation. The blank analysis is typically performed prior to sample analyses, but may be useful if determined between samples or at the end of a sample sequence to provide additional data regarding instrument operation or residual sample carryover from previous sample analyses.

NOTE 3—If automatic baseline correction (see Note 1) is provided by the gas chromatograph, further correction of area slices may not be required. However, if an electronic offset is added to the signal after baseline compensation, additional area slice correction may be required in the form of offset subtraction. Consult the specific instrumentation instructions to determine if an offset is applied to the signal. If the algorithm used is unclear, the slice area data can be examined to determine if further correction is necessary. Determine if any offset has been added to the compensated signal by examining the corrected area slices of those time slices which precede the elution of any chromatographic unretained substance. If these corrected area slices (representing the true baseline) deviate from zero, subtract the average of these corrected area slices from each corrected area slice in the analysis.

9.3 *Retention Time versus Boiling Point Calibration*—A retention time versus boiling point calibration shall be performed on the same day that analyses are performed. Inject an appropriate aliquot (0.2 µL to 2.0 µL) of the calibration mixture (see 7.6) into the chromatograph, using the analysis sequence protocol. Obtain a normal (peak detection) data record in order to determine the peak retention times and the peak areas for each component. Collect a time slice area record if a boiling range distribution report is desired. Fig. 1 illustrates a graphical plot of a calibration analysis.

9.3.1 Inspect the chromatogram of the calibration mixture for evidence of skewed (non-Gaussian shaped) peaks. Skewness is often an indication of overloading the sample capacity of the column, which will result in displacement of the peak apex relative to non-overloaded peaks. Distortion in retention time measurement and hence errors in boiling point temperature calibration will be likely if column overloading occurs. The column liquid phase loading has a direct bearing on acceptable sample size. Reanalyze the calibration mixture using a smaller sample size or a more dilute solution to avoid peak distortion.

9.3.1.1 *Skewness Calculation*—Calculate the ratio A/B on specified peaks in the calibration mixture as indicated by the designations in Fig. 2. A is the width in seconds of the portion of the peak eluting prior to the time of the peak apex and measured at 10 % of peak height (0.10-H), and B is the width in seconds of the portion of the peak eluting after the time of the peak apex at 10 % of peak height (0.10-H). This ratio for *n*-pentacontane (n-C_{50}) peak in the calibration mixture shall not be less than 0.5 nor more than 2.0.

9.3.2 Prepare a calibration table based upon the results of the analysis of the calibration mixture by recording the time of each peak maximum and the boiling point temperature in degrees Celsius (or Fahrenheit) for every component in the mixture. Normal paraffin boiling point temperatures (atmospheric equivalent temperatures) are listed in Table 1. An example of a typical calibration report, showing retention times and boiling points for each *n*-paraffin, is found in Table 3.

9.4 *Sample Preparation*—Sample aliquots are introduced into the gas chromatograph as solutions in a suitable solvent (for example, carbon disulfide or cyclohexane).

9.4.1 Dilute the sample to approximately 2 wt % with the solvent.

9.4.2 Seal (cap) the vial, and mix the contents thoroughly to provide a homogeneous mixture. It may be necessary to warm the mixture initially to effect complete solution of the sample. However, the sample shall be in stable solution at room temperature prior to injection.

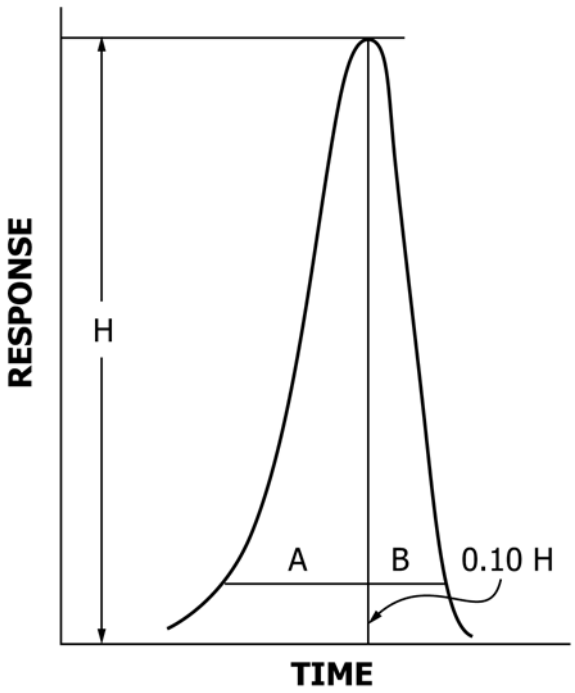

FIG. 2 Designation of Parameters for Calculation of Peak Skewness

TABLE 3 Calibration Table

#	Name	RT (min)	BP (°C)
1	n-C5	0.11	36.1
2	n-C6	0.14	68.7
3	N-C7	0.21	98.4
4	N-C8	0.34	125.7
5	N-C9	0.61	150.8
6	N-C10	1.11	174.1
7	N-C11	1.89	195.9
8	N-C12	2.91	216.3
9	N-C13	4.02	235.4
10	N-C14	5.17	253.9
11	N-C15	6.30	270.6
12	N-C16	7.39	287.2
13	N-C17	8.42	301.9
14	N-C18	9.42	316.1
15	N-C20	11.27	343.9
16	N-C22	12.94	368.3
17	N-C24	14.51	391.1
18	N-C26	15.97	412.2
19	N-C2S	17.33	431.1
20	N-C30	18.60	449.7
21	N-C32	19.79	466.1
22	N-C34	20.92	481.1
23	N-C36	21.98	496.1
24	N-C38	22.98	508.9
25	N-C40	23.96	522.2
26	N-C42	24.84	533.9
27	N-C44	25.70	545.0
28	N-C46	26.52	556.1
29	N-C48	27.30	566.1
30	N-C50	28.06	575.0
31	N-C52	28.78	583.9
32	N-C54	29.48	592.2
33	N-C56	30.15	600.0
34	N-C58	30.81	807.8
35	N-C60	31.47	615.0
36	n-C62	32.06	622.2
37	n-C64	32.65	628.9

9.5 *Sample Analysis*—Using the analysis sequence protocol inject a diluted sample aliquot into the gas chromatograph. Collect a contiguous time slice record of the entire analysis (area slice mode).

9.5.1 Be careful that the injection size chosen does not exceed the linear range of the detector. The typical sample size ranges from 0.2 µL to 2.0 µL of the diluted sample. The maximum sample signal amplitude should not exceed the maximum calibration signal amplitude. A sample chromatogram is found in Fig. 3.

10. Calculations

10.1 *Acquisition Rate Requirements:*

10.1.1 The number of slices required at the beginning of data acquisition depends on chromatographic variables such as the column flow, column film thickness, and initial column temperature as well as column length. In addition, the detector signal level has to be as low as possible at the initial temperature of the analysis. The detector signal level for both the sample signal and the blank at the beginning of the run has to be similar for proper zeroing of the signals.

10.1.2 The sampling frequency has to be adjusted so that at least a significant number of slices are acquired prior to the start of elution of sample or solvent. For example, if the time for start of sample elution is 0.06 min (3.6 s), a sampling rate of 5 Hz would acquire 18 slices. However, a rate of 1 Hz would only acquire 3.6 slices which would not be sufficient for zeroing the signals. Rather than specifying number of slices, it is important to select an initial time segment, that is, one or two seconds. Insure that the smallest number of slices is 5 or greater.

10.1.3 Verify that the slice width used to acquire the sample chromatogram is the same used to acquire the blank run chromatogram.

10.2 *Chromatograms Offset for Sample and Blank*—Perform a slice offset for the sample chromatogram and blank chromatogram. This operation is necessary so that the signal is corrected from its displacement from the origin. This is achieved by determining an average slice offset from the slices accumulated in the first segment (that is, first s) and performing a standard deviation calculation for the first N slices accumulated. It is carried out for both sample signal and baseline signal.

10.2.1 *Sample Offset:*

10.2.1.1 Calculate the average slice offset of sample chromatogram using the first second of acquired slices. Insure that no sample has eluted during this time and that the number of slices acquired is at least 5. Throw out any of the first N slices selected that are not within one standard deviation of the average and recompute the average. This eliminates any area that is due to possible baseline upset from injection.

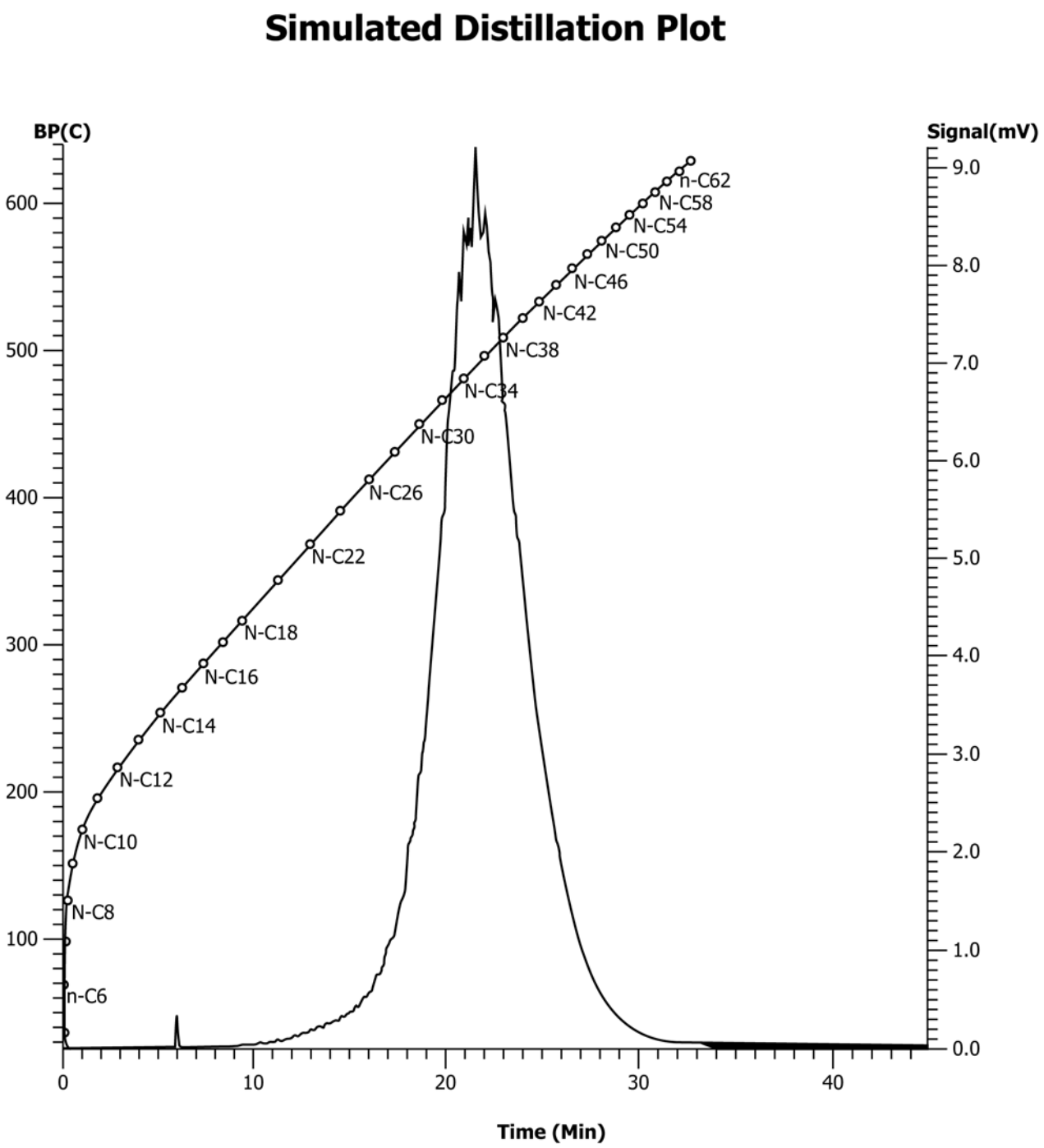

FIG. 3 Typical Chromatogram

10.2.1.2 Subtract the average slice offset from all the slices of the sample chromatogram. Set negative slices to zero. This will zero the chromatogram.

10.2.2 *Blank Offset:*

NOTE 4—If you are using electronic baseline compensation proceed to 10.4. It is strongly recommended that a blank baseline be acquired with or without solvent according to how the sample was prepared for injection. The slice by slice offset is a preferred method for offset the signals.

10.2.2.1 Repeat 10.2.1 using the blank run table.

10.3 *Offset the Sample Chromatogram with Blank Chromatogram*—Subtract from each slice in the sample chromatogram table with its correspondent slice in the blank run chromatogram table. Set negative slices to zero.

10.4 *Determine the Start of Sample Elution Time.*

10.4.1 *Calculate the Total Area*—Add all the corrected slices in the table. If the sample to be analyzed has a solvent peak, start counting area from the point at which the solvent peak has eluted completely. Otherwise, start at the first corrected slice.

10.4.2 *Calculate the Rate of Change Between Each Two Consecutive Area Slices*—Begin at the slice set in 10.4.1 and work forward. The rate of change is obtained by subtracting the area of a slice from the area of the immediately preceding slice and dividing by the slice width. The time where the rate of change first exceeds 0.0001 % per second of the total area (see 10.4.1) is defined as the start of sample elution time. To reduce the possibility of noise or an electronic spike falsely indicating the start of sample elution time, a 1-s slice average can be used instead of a single slice. For noisier baselines, a slice average larger than 3 s may be required

10.5 *Determine the End of Sample Elution Time.*

10.5.1 *Calculate the Rate of Change Between Each Two Consecutive Area Slices*—Begin at the end of run and work backwards. The rate of change is obtained by subtracting the area of a slice from the area of the immediately preceding slice and dividing by the slice width. The time where the rate of change first exceeds 0.00001 % per second of the total area (see 10.4.1) is defined as the end of sample elution time. To reduce the possibility of noise or an electronic spike falsely indicating the end of sample elution time, a 1-s slice average can be used instead of a single slice. For noisier baselines, a slice average larger than 1 s may be required.

10.6 *Calculate the Sample Total Area*—Add all the slices from the slice corresponding to the start of sample elution time to the slice corresponding to the end of sample elution time.

10.7 *Normalize to Area Percent*—Divide each slice in the sample chromatogram table by the total area (see 10.6) and multiply it by 100.

10.8 *Calculate the Boiling Point Distribution Table:*

10.8.1 *Initial Boiling Point*—Add slices in the sample chromatogram until the sum is equal to or greater than 0.5 %. If the sum is greater than 0.5 %, interpolate (refer to the algorithm in 10.9.1) to determine the time that will generate the exact 0.5 % of the area. Calculate the boiling point temperature corresponding to this slice time using the calibration table. Use interpolation when required (refer to the algorithm in 10.9.2).

10.8.2 *Final Boiling Point*—Add slices in the sample chromatogram until the sum is equal to or greater than 99.5 %. If the sum is greater than 99.5 %, interpolate (refer to the algorithm in 10.9.1) to determine the time that will generate the exact 99.5 % of the area. Calculate the boiling point temperature corresponding to this slice time using the calibration table. Use interpolation when required (refer to the algorithm in 10.9.2).

10.8.3 *Intermediate Boiling Point*—For each point between 1 % and 99 %, find the time where the cumulative sum is equal to or greater than the area percent being analyzed. As in 10.8.1 and 10.8.2, use interpolation when the accumulated sum exceeds the area percent to be estimated (refer to the algorithm in 10.9.1). Use the calibration table to assign the boiling point.

10.9 *Calculation Algorithm:*

10.9.1 Calculations to determine the exact point in time that will generate the X percent of total area, where $X = 0.5, 1, 2, ..., 99.5$ %.

10.9.1.1 Record the time of the slice just prior to the slice that will generate an accumulative slice area larger than the X percent of the total area. Let us call this time, T_s, and the accumulative area at this point, A_c.

10.9.1.2 Calculate the fraction of the slice required to produce the exact X percent of the total area:

$$A_x = \frac{X - A_c}{A_{c+1} - A_c} \tag{3}$$

where:

A_x = fraction of the slice that will yield the exact percent,
A_c = cumulative percent up to the slice prior to X,
A_{c+1} = cumulative percent up to the slice right after X, and
X = desired cumulative percent.

10.9.1.3 Calculate the time required to generate the fraction of area A_x:

$$T_f = A_x \cdot W \tag{4}$$

where:

W = the slice width,
A_x = fraction of the slice that will yield the exact percent, and
T_f = fraction of time that will yield A_x.

10.9.1.4 Record the exact time where the accumulative area is equal to the X percent of the total area:

$$T_t = T_s + T_f \tag{5}$$

where:

T_s = fraction of the slice that yields the cumulative percent up to the slice prior to X,
T_f = fraction of time that will yield A_x, and
T_t = time where the cumulative area is equal to X percent of the total area.

10.9.2 Interpolate to determine the exact boiling point given the retention time corresponding to the cumulative slice area.

10.9.2.1 Compare the given time against each retention time in the calibration table. Select the nearest standard having a retention time equal to or larger than the interpolation time. (**Warning—**The retention time table shall be sorted in ascending order.)

10.9.2.2 If the interpolation time is equal to the retention time of the standard, record the corresponding boiling point.

10.9.2.3 If the retention time is not equal to a retention time of the standards (see 9.3), interpolate the boiling point temperature as follows:

10.9.2.4 If the interpolation time is less than the first retention time in the calibration table, then extrapolate using the first two components in the table:

$$BP_x = m_1 \cdot (RT_x - RT_1) + BP_1 \tag{6}$$

where:

m_1 = $(BP_2 - BP_1) / (RT_2 - RT_1)$,
BP_x = boiling point extrapolated,
RT_x = retention time to be extrapolated,

RT_1 = retention time of the first component in the calibration table,
BP_1 = boiling point of the first component in the calibration table,
RT_2 = retention time of the second component in the calibration table, and
BP_2 = boiling point of the second component in the calibration table.

10.9.2.5 If the interpolation time is between two retention times in the calibration table, then interpolate using the upper and lower standard components:

$$BP_x = m_u \cdot (RT_x - RT_1) + BP_1 \quad (7)$$

where:

m_u = $(BP_u - BP_1) / (RT_u - RT_1)$,
BP_x = boiling point extrapolated,
RT_x = retention time to be extrapolated,
RT_1 = retention time of the lower bound component in the calibration table,
BP_1 = boiling point of the lower bound component in the calibration table,
RT_u = retention time of the upper bound component in the calibration table, and
BP_u = boiling point of the upper bound component in the calibration table.

10.9.2.6 If the interpolation time is larger than the last retention time in the calibration table, then extrapolate using the last two standard components in the table:

$$BP_x = m_n \cdot (RT_x - RT_{n-1}) + BP_{n-1} \quad (8)$$

where:

m_n = $(BP_n - BP_{n-1}) / (RT_n - RT_{n-1})$
BP_x = boiling point extrapolated,
RT_x = retention time to be extrapolated,
RT_{n-1} = retention time of the standard component eluting prior to the last component in the calibration table,
BP_{n-1} = boiling point of the standard component eluting prior to the last component in the calibration table,
RT_n = retention time of the last component in the calibration table, and
BP_n = boiling point of the standard component in the calibration table.

11. Report

11.1 Report the temperature to the nearest 0.5 °C (1 °F) at 1 % intervals between 1 % and 99 % and at the IBP (0.5 %) and the FBP (99.5 %). Other report formats based upon users' needs may be employed.

NOTE 5—If a plot of the boiling point distribution curve is desired, use a spreadsheet with uniform subdivisions and use either retention time or temperature as the horizontal axis. The vertical axis will represent the sample boiling range distribution from 0 % to 100 %. Plot each boiling point temperature against its corresponding accumulated percent slice area. Draw a smooth curve connecting the points.

12. Precision and Bias[5]

12.1 *Precision*—The precision of this test method as determined by the statistical examination of the Interlaboratory Study (ILS) is as follows:

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1725.

TABLE 4 Repeatability (r) and Reproducibility (R)

X = Temperature Obtained in (°C)[A]			
% m/m	r (°C)	R (°C)	(°C) Temperature Range RR Samples
0.5 (IBP)	2.2844	9.3886	142 to 419
5	1.4949E03 * X ^ (-1.1741)	4.6038E03 * X ^ (-1.1741)	201 to 458
10	1.6442	4.2859	232 to 465
20	8.753E-03 * (588.151 - X)	2.152E-02 * (588.151 - X)	272 to 487
30	6.157E-03 * (637.8967 - X)	1.835E-02 * (637.8967 - X)	303 to 497
40	1.114E-02 * (568.737 - X)	2.956E-02 * (568.737- X)	329 to 505
50	1.2821	3.3963	354 to 512
60	1.2853	2.8556	382 to 519
70	1.0698 * (528.001 - X) ^ 0.0664	2.367 * (528.001 - X) ^ 0.0664	414 to 526
80	1.5749	3.0534	435 to 536
90	2.4209 *(560.6001 - X) ^ -0.1658	4.9104* (560.6001 - X) ^ -0.1658	449 to 557
95	1.8112	5.0888	462 to 563
99.5 (FBP)	7.259E-04 * X ^ 1.4248	2.517E-03 * X ^ 1.4248	508 to 612

[A] Round boiling point to the nearest 0.5 °C.

12.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values by only one case in twenty (see Table 4).

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only one case in twenty (see Table 4).

12.1.3 Example calculations on the precision is shown in Table 5.

NOTE 6—This precision estimate is based on the analysis of 10 samples by 10 laboratories.

12.2 *Bias*—Because the boiling point distribution can be defined only in terms of a test method, no bias for these procedures in Test Method D7213 for determining the boiling range distribution of light and middle petroleum fractions by gas chromatography have been determined.

12.2.1 A rigorous, theoretical definition of the boiling range distribution of petroleum fractions is not possible due to the complexity of the mixture as well as the unquantifiable interactions among the components (for example, azeotropic behavior). Any other means used to define the distribution would require the use of a physical process, such as conventional physical distillation or gas chromatographic characterization. This would therefore result in a method-dependent definition and would not constitute a true value from which bias can be calculated.

13. Keywords

13.1 boiling range distribution; distillation; gas chromatography; petroleum; simulated distillation

TABLE 5 Example of Precision at Selected Temperatures

% m/m	T (°C)	r (°C)	R (°C)
0.5 (IBP)	280.5	2.3	9.4
5	329.5	1.7	5.1
10	348.5	1.6	4.3
20	379.5	1.8	4.5
30	400	1.5	4.4
40	435.5	1.5	3.9
50	433	1.3	3.4
60	450.5	1.3	2.9
70	470	1.4	3.1
80	485.5	1.6	3.0
90	503	1.2	2.5
95	512.5	1.8	5.0
99.5 (FBP)	560	6.0	20.7

APPENDIXES

(Nonmandatory Information)

X1. BOILING POINTS OF NONPARAFFINIC HYDROCARBONS

X1.1 There is an apparent discrepancy in the boiling point of multiple ring-type compounds. When the retention time of these compounds are compared to *n*-paraffins of equivalent atmospheric boiling point, these ring compounds appear to be eluted early from methyl silicone columns. A plot showing 36 compounds other than *n*-paraffins plotted along the calibration curve for *n*-paraffins alone is shown in Fig. X1.1. The numbered dots are identified in Table X1.1. In this figure the atmospheric boiling points are plotted against the observed retention times. If columns contained different percentages of stationary phase or different temperature programming rates are used, the slope and curvature on the *n*-paraffin curve (solid line) would change, but the relative relationships would remain essentially the same. Deviations of simulated distillation boiling points, as estimated from the curve, from actual boiling points for a few compounds are shown in Table X1.2. The deviations obtained by plotting boiling points at 10 mm Hg rather than 76 mm Hg are tabulated also. It is apparent that the deviation is much less at 10 mm Hg pressure. This indicates that the distillation data produced by gas chromatography closely approximates those obtained in reduced pressure distillations. Since the vapor-pressure-temperature curves for multiple-ring type compounds do not have the same slope or curvature as those of *n*-paraffins, an apparent discrepancy would exist when *n*-paraffin boiling points at atmospheric pressure are used.

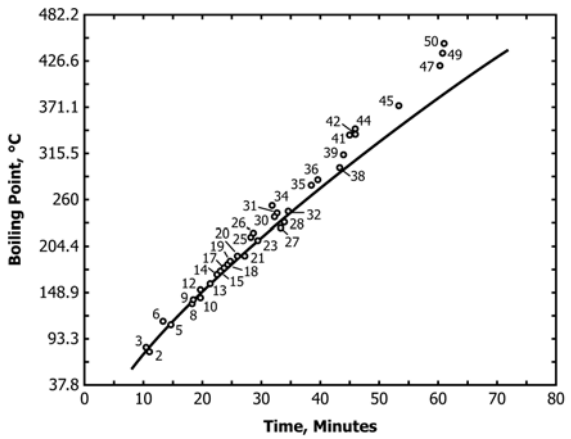

FIG. X1.1 Boiling Point—Retention Time Relationships for Several High-Boiling Multiple-Ring Type Compounds (see Table X1.1)

X1.2 However, this discrepancy does not introduce any significant error when comparing with laboratory distillation because the pressure shall be reduced in such procedures when overhead temperature reach approximately 260 °C (500 °F) to prevent cracking of the sample. Thus, distillation data are subject to the same deviations experienced in simulated distillation by gas chromatography.

TABLE X1.1 Compound Identification—Number Dots (see Fig. X1.1)

Number	Boiling Point, °C (°F)	Compound	Number	Boiling Point, °C (°F)	Compound
2	80 (176)	benzene	27	227 (441)	di-*n*-amylsulfide
3	84 (183)	thiophene	28	234 (453)	tri-isopropylbenzene
5	111 (231)	toluene	30	241 (466)	2-methlynaphthalene
6	116 (240)	pyridine	31	295 (473)	1-methlynaphthalene
8	136 (277)	2,5-dimethlythiophene			
9	139 (282)	*p*-xylene	34	254 (894)	indole
10	143 (289)	di-*n*-propylsulfide	35	279 (534)	acenaphthene
12	152 (306)	cumene			
13	159 (319)	1-hexahydroindan	38	298 (568)	*n*-decylbenzene
14	171 (339)	1-decene	39	314 (598)	1-octadecene
15	173 (344)	*sec*-butylbenzene			
17	178 (352)	2,3-dihydroindene	41	339 (642)	phenanthrene
18	183 (361)	*n*-butylbenzene	42	342 (647)	anthracene
19	186 (366)	trans-decalin			
20	194 (382)	cis-decalin	44	346 (655)	acridine
21	195 (383)	di-*n*-propyldisulfide	45	395 (743)	pyrene
23	231 (416)	1-dodecene	47	404 (496)	triphenylene
25	218 (424)	naphthalene	49	438 (820)	naphthacene
26	221 (430)	2,3-benzothiophene	50	447 (837)	chrysene

TABLE X1.2 Deviations of Simulated Distillation Boiling Points from Actual Boiling Points

Compound	Boiling Point, °C (°F) (760 mm)	Deviations from Actual Boiling Point, °C (°F) (760 mm)	Deviations from Actual Boiling Point, °C (°F) (10 mm)
benzene	80 (176)	+3 (+6)	-2 (-4)
thiophene	84 (183)	+4 (+7)	+1 (+2)
toluene	111 (231)	+2 (+3)	-1 (-2)
p-xylene	139 (282)	0 (0)	+2 (+4)
1-dodecene	213 (416)	0 (0)	0 (0)
naththalene	218 (424)	-11 (-20)	-4 (-8)
2,3-benzothiophene	221 (430)	-13 (-23)	0 (0)
2-methylnaphthalene	241 (466)	-12 (-21)	-2 (-3)
1-methylnaphthalene	245 (473)	-12 (-21)	-1 (-1)
dibenzothiophene	332 (630)	-32 (-58)	-6 (-10)
phenanthrene	339 (642)	-35 (-63)	-9 (-16)
athracene	342 (647)	-36 (-64)	-8 (-15)
pyrene	395 (743)	-48 (-87)	-16 (-29)
chrysene	447 (837)	-60 (-108)	[A]

[A] No data at 10 mm for chrysene.

X2. ALTERNATIVE CARRIER GASES TO HELIUM

NOTE X2.1—This appendix contains instrument conditions and results obtained using nitrogen or hydrogen as an alternate carrier gas. At this time, because the test method precision and bias performance information using the alternate carrier gases and conditions listed in this appendix have not been studied in accordance with the proper ASTM ILS process, this appendix is included only for information purposes. Results obtained under the conditions described in this appendix are not considered to be valid D2887 results, and shall not be represented as such. (**Warning—** Use caution when hydrogen is used as the carrier gas. The use of a hydrogen sensor in the GC oven is strongly recommended in order to shut off the hydrogen source in case of a high concentration buildup of hydrogen which exceeds the explosive limit.)

X2.1 This section lists the conditions for using alternative carrier gases to helium. The data below shows the conditions:

D7213 NITROGEN
Column: 5 m 0.5 3 mm-0.8 µ PDMS
Carrier: N_2 20 mL/min
Oven: 40 °C to 390 °C at 10 °C/min
hold -10 min
FID: 400 °C
Air: 450 mL/min H_2: 45 mL/min
Mup: N_2: 15 mL/min
SAMPLE: 0.1 µL 10 % to 20 % IN CS_2

D7213 HYDROGEN
Column: 5 m 0.53 mm 0.8 µ PDMS
Carrier: H_2 20 mL/min
Oven: 40 °C to 390 °C at 10 °C/min
hold-5 min
FID: 400 °C
Air: 450 mL/min H_2: 25 mL/min
Mup: N_2: 25 mL/min
SAMPLE : 0.1 µL 10 % to 20 % IN CS_2

X2.2 Typical chromatogram obtained with hydrogen carrier for the calibration is shown in Fig. X2.1. Fig. X2.2 is the chromatogram of the reference gas oil #1–batch 2 utilizing H_2 as carrier.

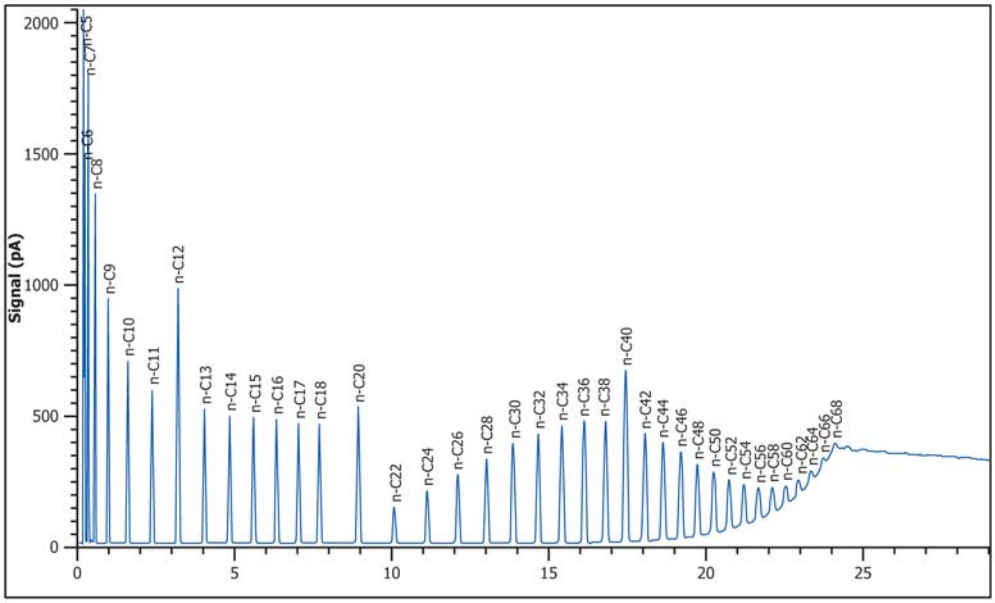

FIG. X2.1 Typical Chromatogram Obtained with Hydrogen Carrier for Calibration

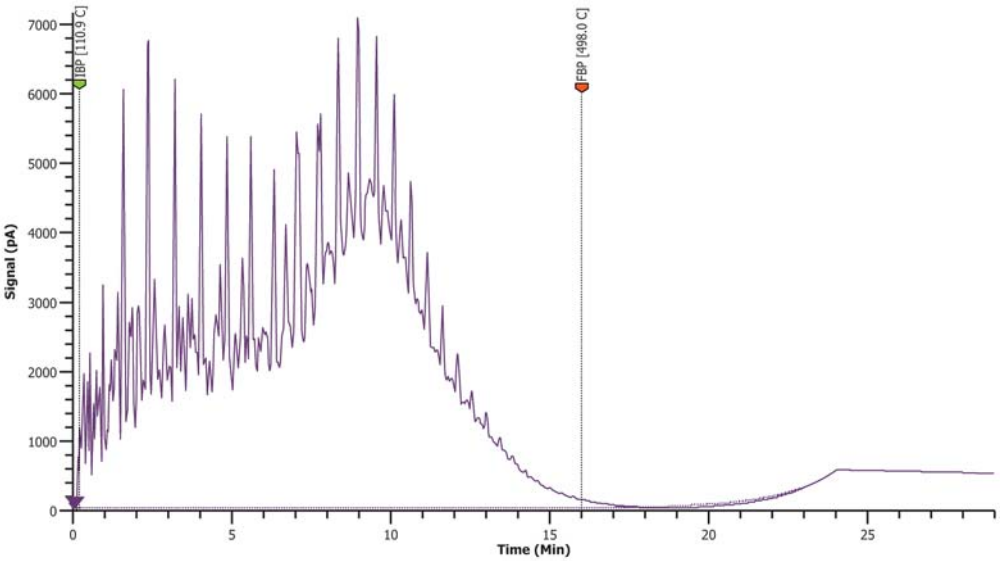

FIG. X2.2 Chromatogram of Reference Gas Oil #1–Batch 2 Utilizing H_2 as Carrier

TABLE X2.1 ASTM D2887 Reference Gas Oil #1 Batch 2 Boiling Point Distribution Values Obtained with H_2 (left) and with N_2 (right) Carrier Gases

% Off	BP(°C)	QC(°C)	(-)	Limit	% Off	BP(°C)	QC(°C)	(-)	Limit
IBP	110.9	106.1	4.8	7	IBP	112.4	106.1	6.3	7
5	174.2	172.8	1.4	4.1	5	174.7	172.8	1.9	4.1
10	196.7	195.6	1.2	4.4	10	197.7	195.6	2.2	4.4
15	216.8	215.6	1.3	4.7	15	217.8	215.6	2.2	4.7
20	234.4	233.3	1	5	20	235.4	233.3	2.1	5
25	251.2				25	252.7			
30	267.5	266.7	0.8	4.8	30	268.9	266.7	2.3	4.8
35	283.4				35	284.9			
40	298.1	297.8	0.3	4.3	40	299.3	297.8	1.5	4.3
45	310.2				45	311.1			
50	320.8	321.1	–0.3	4.3	50	321.8	321.1	0.7	4.3
55	331.4				55	332.2			
60	341.1	341.7	–0.6	4.3	60	342	341.7	0.3	4.3
65	349.9	350	–0.1	4.3	65	350.6	350	0.6	4.3
70	358.1	358.3	–0.3	4.3	70	358.8	358.3	0.5	4.3
75	367.5	367.8	–0.3	4.3	75	368.1	367.8	0.4	4.3
80	377.4	377.8	–0.4	4.3	80	378.1	377.8	0.3	4.3
85	389.9	390	–0.1	4.3	85	390.6	390	0.6	4.3
90	406.3	406.1	0.2	4.3	90	407	406.1	0.9	4.3
95	430.9	431.1	–0.2	5	95	431.9	431.1	0.8	5
FBP	498	496.1	1.9	11.8	FBP	499.7	496.1	3.6	11.8

SUMMARY OF CHANGES

Subcommittee D02.04.0H has identified the location of selected changes to this standard since the last issue (D7213 – 14) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Revised Note X2.1.

Subcommittee D02.04.0H has identified the location of selected changes to this standard since the last issue (D7213 – 11) that may impact the use of this standard. (Approved Oct. 1, 2014.)

(1) Added new Appendix X2.