Designation: D7500 – 15

Standard 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[1]

This standard is issued under the fixed designation D7500; 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 by capillary gas chromatography using flame ionization detection. This standard test method has been developed through the harmonization of two test methods, Test Method D6352 and IP 480. As both of these methods cover the same scope and include very similar operating conditions, it was agreed that a single standard method would benefit the global simulated distillation community.

1.2 This test method is not applicable for the analysis of petroleum or petroleum products containing low molecular weight components (for example naphthas, reformates, gasolines, diesel). Components containing hetero atoms (for example alcohols, ethers, acids, or esters) or residue are not to be analyzed by this test method. See Test Methods D7096, D2887, or D7213 for possible applicability to analysis of these types of materials. This method is also not suitable for samples that will not elute completely from the gas chromatographic column, leaving residues. For such samples as crude oils and residues, see Test Methods D5307 and D7169.

1.3 This test method is applicable to distillates with initial boiling points above 100 °C and final boiling points below 735 °C (carbon 110); for example, distillates (IBP > 100 °C), base oils and lubricating base stocks.

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
D5307 Test Method for Determination of Boiling Range Distribution of Crude Petroleum by Gas Chromatography (Withdrawn 2011)[3]
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
D7213 Test Method for Boiling Range Distribution of Petroleum Distillates in the Boiling Range from 100 °C to 615 °C by 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

2.2 *ISO Standard:*[4]

ISO 3170 Petroleum Liquids Manual Sampling

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[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.

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

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

3. Terminology

3.1 *Definitions*—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. For definitions of these terms used in this test method, refer to 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 6.4.1), 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 % 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 frequency used in sampling (analog) the chromatographic detector signal during an analysis. The slice rate is expressed in Hz (for example integrations or slices per second).

3.2.7 *slice time, n*—the inverse function of the acquisition rate. It is the time duration of each sampling pulse usually expressed in seconds. 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 area point to the final area point, where the chromatographic signal has returned to baseline after complete sample elution.

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 n-C_{10} for normal-decane, i-C_{14} for *iso*-tetradecane).

4. Summary of Test Method

4.1 The boiling range distribution determination by distillation is simulated by the use of gas chromatography. 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 specified linear rate to affect 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 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 the test method, 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 medium and heavy petroleum distillate fractions provides an insight into the composition of feed stocks and products related to petroleum refining processes (for example, hydrocracking, hydrotreating, visbreaking, or deasphalting). The gas chromatographic simulation of this determination 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 distillates (IBP > 100 °C) and heavy petroleum distillate fractions beyond the scope of Test Method D2887 (538 °C).

5.3 Boiling range distributions obtained by this test method have not been analyzed for correlation to those obtained by low efficiency distillation, such as with Test Method D86 or D1160. This test method does not claim agreement between these physical distillations and simulated distillation. Efforts to resolve this question will continue. When successful resolutions of the questions are determined, this test method will be revised accordingly.

6. Apparatus

6.1 *Chromatograph*—The gas chromatographic system used shall have the following performance characteristics:

6.1.1 *Carrier Gas Flow Control*—The chromatograph shall be equipped with carrier gas pressure or flow control capable of maintaining constant carrier gas flow to ±1 % throughout the column temperature program cycle.

6.1.2 *Column Oven*—Capable of sustained and linear programmed temperature operation from near ambient (for example, 30 °C to 35 °C) up to 430 °C.

6.1.3 *Column Temperature Programmer*—The chromatograph shall be capable of linear programmed temperature operation up to 430 °C at selectable linear rates up to 10 °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.

6.1.4 *Detector*—This test method requires the use of a flame ionization detector (FID). The detector shall meet or exceed the following specifications in accordance with Practice E594. Check the detector according the instrument manufacturers instructions.

6.1.4.1 *Operating Temperature*—100 °C to 430 °C.

6.1.4.2 Connection of the column to the detector shall be such that no temperature below the column temperature exists between the column and the detector. Refer to Practice E1510 for proper installation and conditioning of the capillary column.

6.1.5 *Sample Inlet System*—Any sample inlet system capable of meeting the performance specification in Annex A3 and execute the conditions of Table 2. Programmable temperature vaporization (PTV) and cool on-column (COC) injection systems have been used successfully.

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 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. Fused silica (aluminum coated) and stainless steel columns with 0.53 mm to 0.75 mm internal diameter have been successfully used. Cross-linked or bonded 100 % dimethyl-polysiloxane stationary phases with film thickness of 0.09 µm to 0.17 µm have been used. The column length and liquid phase film thickness shall allow the elution of C_{110} *n*-paraffin (BP = 735 °C). The column and conditions shall provide separation of typical petroleum hydrocarbons in order of increasing boiling point and meet the column performance requirements of A3.2.1. The column shall provide a resolution not less than 2 and not higher than 4 using the test method operating conditions in Table 2.

6.4 *Data Acquisition System:*

6.4.1 *Integrator/Computer System*—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 processing mode). In addition, the system shall be capable of converting the continuously integrated detector signal into area slices of fixed duration (slice mode). These contiguous area slices, collected for the entire analysis, are stored for later processing. A similar collection of contiguous slices is also collected for the blank run. It is necessary that the number of slices collected for sample and blank analysis are the same. 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.

TABLE 1 Reference Material 5010[A]

% Dist. m/m	°C	°F	r, °C	R, °C	r, °F	R, °F
IBP	421	789	3	9	5	16
5	476	888	2	4	4	8
10	491	916	2	4	3	7
20	510	950	2	5	3	9
30	524	975	2	5	3	9
40	536	998	2	5	3	9
50	548	1018	2	5	3	9
60	559	1039	2	5	3	9
70	572	1061	2	5	3	9
80	585	1085	2	5	3	9
90	602	1116	2	5	3	9
95	617	1142	2	5	3	9
FBP	661	1223	9	17	16	31

[A] Values obtained from including Reference Oil 5010 in the ILS sample set.

TABLE 2 Typical Operating Conditions for Gas Chromatograph

Column length, m	5
Column internal diameter, mm	0.53
Column material	Metal
Stationary phase type	methyl silicone
Film thickness, µm	0.09 to 0.17
Initial column temperature, °C	35
Initial hold time, min	0
Final column temperature, °C	430
Final hold time, min	10
Program rate, °C/min	10
Injector initial temperature, °C	100
Injector final temperature, °C	430
Injector program rate, °C/min	15
Detector temperature, °C	450
Make-up gas flow, He or N2, mL/min[A]	20
Hydrogen Flow, mL/min[A]	45
Air Flow, mL/min[A]	450
Carrier gas	He
Carrier gas flow rate, constant flow, mL/min	19
Sample size, µL[A,B]	1.0
Sample concentration, % (m/m)	2
Injector	PTV or COC

[A] Consult with the manufacturer's operations manual.

[B] Monitor skewness when varying the injection volume.

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 *Liquid Stationary Phase*—A methyl silicone stationary phase for the column.

7.2 *Carrier Gases*—Helium, of at least 99.999 % (v/v) purity. Any oxygen present is removed by a chemical resin filter. (**Warning—**Follow the safety instructions from the filter supplier.) Total impurities not to exceed 10 mL/m^3. Helium or Nitrogen (99.999 %) can also be used as detector makeup gas. The use of alternative carrier gases hydrogen and nitrogen is described in Appendix X1.

7.3 *Hydrogen*—99.999 % Grade suitable for flame ionization detectors. Total impurities not to exceed 10 mL/m^3.

7.4 *Compressed Air*—Regulated for flame ionization detectors. Total impurities not to exceed 10 mL/m^3.

7.5 *Alkanes*—Normal alkanes of at least 98 % (m/m) purity from C_5 to C_{10}, C_{12}, C_{14}, C_{16}, C_{18}, C_{20}, C_{24}, C_{26}, and C_{28}, are to be used with Polywax 655 or 1000.[5] A solution of these alkanes is prepared by adding 500 mg of each alkane into a 20 mL vial. Additionally *n*-tetracontane (C_{40}) can also be added to for ease of carbon counting. This solution is used to spike the Polywax[5] solution.

7.6 *Polywax 655 or 1000.*[5]

7.7 *Carbon Disulfide*—Purity 99.7 % (v/v) minimum. (**Warning—**Extremely flammable and toxic by inhalation.)

7.8 *Calibration Mix*—A suitable calibration mixture can be obtained by dissolving a hydrogenated polyethylene wax (for example, Polywax 655[5] or Polywax 1000[5]) in a volatile solvent (for example, CS_2 or cyclohexane). Solutions of 1 part Polywax[5] to 200 parts solvent can be prepared. Lower boiling point paraffins will have to be added to as specified in 7.5. Fig. 5 illustrates a typical calibration mixture chromatogram. The calibration mix is used to determine the column resolution, skewness of components, and retention time versus boiling point calibration curve. Add 10 µL of the mixture of alkanes prepared in 7.5.

NOTE 2—Commercially available alkane standards are suitable for column performance checks.

NOTE 3—Calibration mixtures are commercially available.

7.9 *Reference Oil 5010*—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 given Table 1.

NOTE 4—The 5010 reference oil is available commercially.

7.10 *Cyclohexane* (C_6H_{12})—(99+ % pure) if necessary, use in place of CS_2 for the preparation of the calibration mixture.

7.11 A Gravimetric blend consisting of 2 distillation fractions is used for system performance check (see A3.3).

8. Sampling and Sample Preparation

8.1 Unless otherwise specified, obtain the laboratory samples by the procedures specified in ISO 3170 and place in glass or metal containers. Do not use plastic containers for sample storage to avoid contamination of the sample because of possible leaching of the plasticizer.

8.2 *Sample Preparation*—Sample aliquots are introduced into the gas chromatograph as solutions in a suitable solvent (for example, CS_2).

8.3 Place approximately 0.1 g to 1 g of the sample aliquot into a screw-capped or crimp-cap vial.

8.4 Dilute the sample aliquot to approximately 1 weight percent to 3 weight percent with the solvent, depending on the boiling point distribution.

8.5 Seal (cap) the vial, and mix the contents thoroughly to provide a homogeneous mixture. Warm the vial if necessary initially to affect complete solution of the sample. Inspect the sample to ensure it is in stable solution at room temperature prior to injection. If necessary, prepare a more dilute solution.

9. Preparation of Apparatus

9.1 *Gas Chromatograph Setup*—Set up and operate the gas chromatograph in accordance with the manufacturer's instructions.

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[5] Polywax is a registered trademark of Baker Petrolite, 12645 West Airport Blvd., Sugar Land, TX 77478.

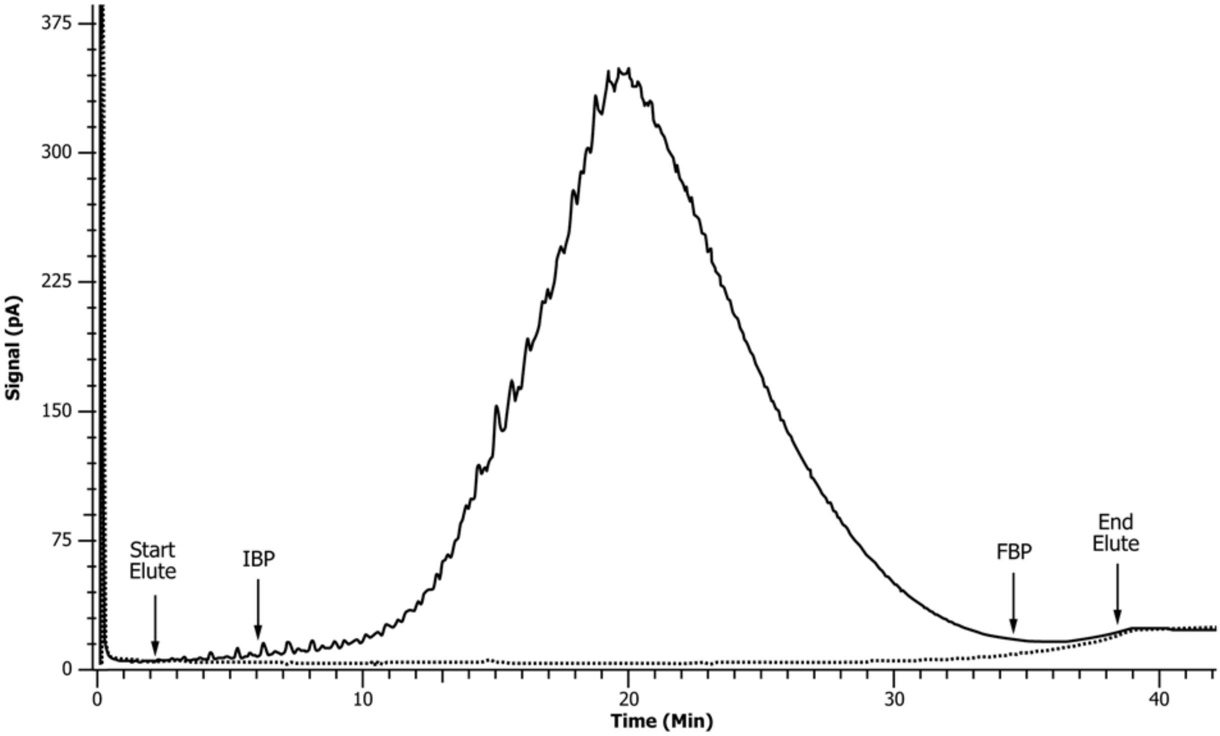

FIG. 1 Typical Sample Chromatogram which has a FBP of 700 ºC

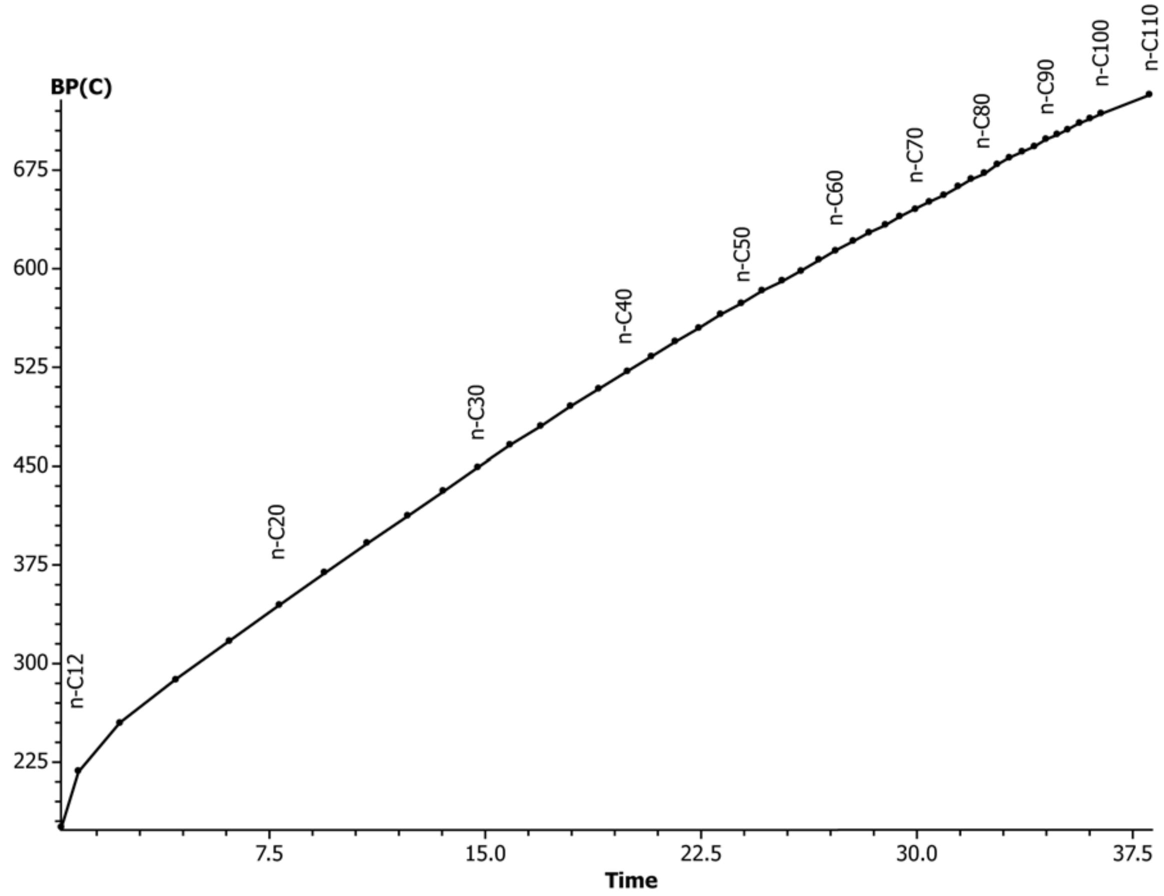

FIG. 2 Typical Calibration Curve of Retention Time versus Boiling Point

NOTE 5—Typical operating conditions are shown in Table 2.

9.2 A new column will require conditioning at the upper test method operating temperature to reduce or eliminate significant liquid phase bleed so that a stable and repeatable chromatographic baseline can be generated. Disconnecting the column will require conditioning prior to calibration and analysis.

9.3 The inlet liner (PTV) and or the initial section of the column (COC and PTV) shall be periodically inspected and replaced in order to remove extraneous deposit or sample residue.

9.4 Perform a blank analysis after a new septum is installed to ensure that no extraneous peaks are produced by the septum. The blank analysis shall be carried out whenever the column is disconnected from carrier flow.

9.5 Ensure that the system's return to baseline is achieved near the end of the run and that the baseline shows no drift at the final isothermal oven temperature.

9.6 Inspect and clean the jet periodically to avoid deposits that form on the jet from combustion of decomposition products from the column liquid stationary phase. These deposits will affect the characteristics of the detector response.

NOTE 6—The following parameters are affected by deposits on the jet: increase in inlet pressure, FID difficulty in lighting, increase in the CS_2 response, and off-specification reference oil. To clean the jet, place it in an ultrasonic cleaner with a suitable solvent and use a cleaning wire if necessary to remove column deposits.

9.7 Check the system performance requirements at installation and at the intervals given and by the procedures specified in Annex A3 with regards to frequency of calibration, check column resolution, peak skewness and verify the detector response with the gravimetric blend.

10. Calibration

10.1 The first run of the day shall be a clean up run and not a usable blank because of the possible elution of extraneous components that have concentrated in the inlet while the instrument is idle. However, a retention time calibration mix (7.8) can be used as first injection.

10.2 Run the calibration mixture (7.8) and confirm the elution of C_{110} within the oven temperature program.

NOTE 7—When C_{110} does not elute within the temperature program, it is recommended to shorten the column. See manufacturer's instructions.

10.3 Ensure the injection volume (or sample concentration) chosen does not allow any peak to exceed the linear range of the detector or overload the column. The skewness of all peaks shall be maintained between 0.8 to 1.8. Values greater than 1.8 indicate the sample is too concentrated and a skew less than 0.8 indicate severe tailing due to an old column or dirty liner or a poorly focused sample. As a guide, 0.2 µL to 1.0 µL of the calibration mixture (7.8) has been found to be suitable for

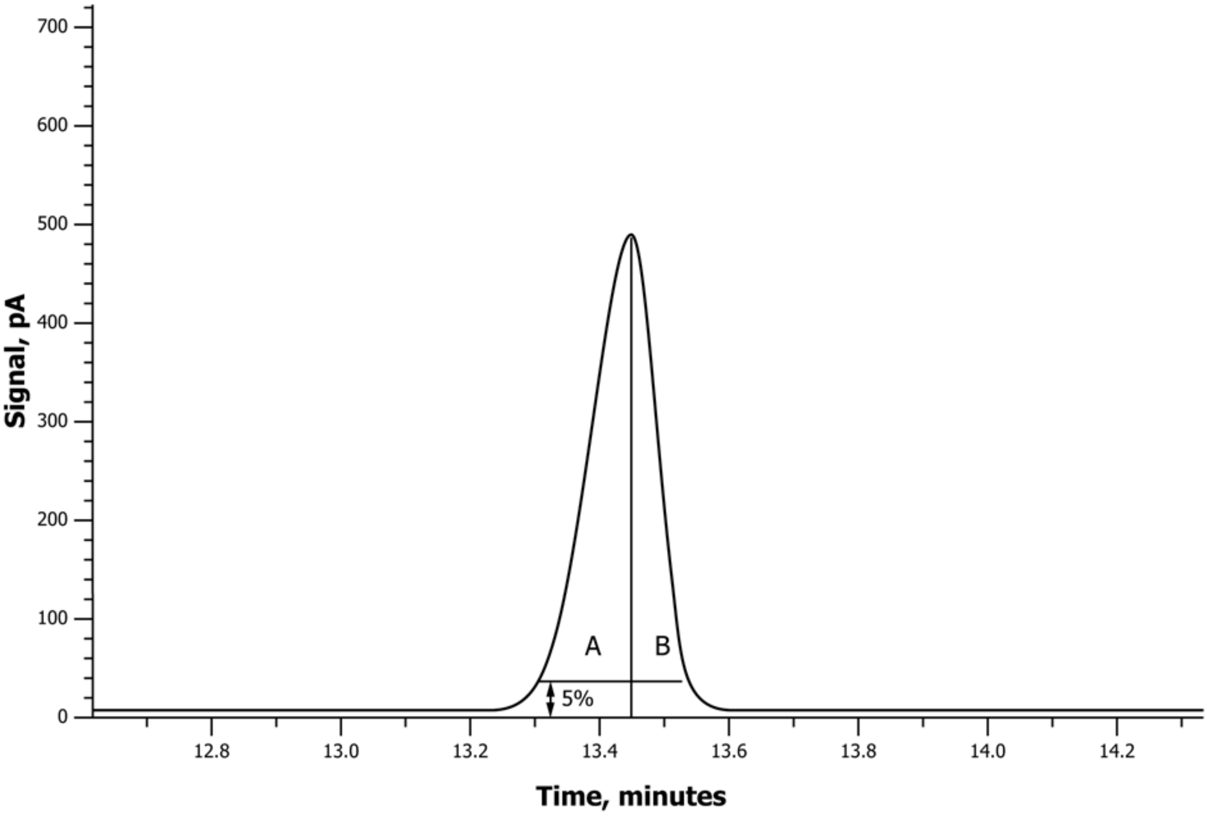

FIG. 3 Peak Skewness for Calibration Mix Peak C_{20}

columns with a film thickness ranging from 0.09 µm to 0.17 µm or less. (See A3.4.)

10.4 Record the retention time of each component and plot the retention time versus the atmospheric boiling point for each component using the boiling points from Table 4. Typical results of the calibration are shown in Table 5 and Fig. 2.

10.5 Inject the Reference Oil 5010 (7.9) using the specified procedure (Section 11). Visually inspect the chromatogram. Using the data system, load the chromatogram (Fig. 6) of the reference oil 5010 and overlay the blank baseline. Enlarge the section of the chromatogram at the end of sample elution and compare the relative magnitudes of the sample and blank baseline with the examples shown in Fig. 7. Ensure that the end of the run merges with the sample chromatogram as shown in Fig. 7. Calculate the boiling range distribution of the reference material by the procedures specified in Annex A1 and compare this with the consensus values for the reference material used as listed in Table 1.

NOTE 8—Fig. 6 shows a typical chromatogram of the 5010 reference oil. Table 6 shows typical boiling point values obtained for the reference oil.

10.6 If the consensus values as shown in Table 1 are not met, check that all hardware is operating properly and all instrument settings are as recommended by the manufacturer. Rerun the retention boiling point calibration as described in 10.3.

11. Sample Analyses Procedure

11.1 Run a solvent (blank) baseline analysis before the first sample analysis and then after every five samples.

11.2 Inspect the baseline at the end of the run for each solvent (blank) injected to ensure that it is constant and stable and is void of extraneous peaks.

NOTE 9—The identification of a constant baseline at the end of the run is critical to the analysis. Constant attention should be given to all factors that influence baseline stability, for example, column substrate bleed, septum bleed, and detector temperature control, constancy of carrier gas flow, leaks, and instrument drift.

11.3 Prepare a Sequence analysis listing all samples and blank to be injected as described in 11.1.

NOTE 10—A sequence is a series of analysis. The length of the sequence depends on the system stability.

11.4 Cool the column and inlet to the starting temperature and inject the selected sample volume.

11.5 Immediately start programming the column temperature and the temperature of the PTV or COC inlet.

11.6 Visually inspect the chromatogram. Using the data system, load each sample chromatogram overlay the nearest blank baseline obtained after the sample as listed in the Sequence. Enlarge the section of the chromatogram at the end of sample elution and compare the relative magnitudes of the sample and blank baseline with the examples shown in Fig. 7. Insure that the end of the run merges with the sample

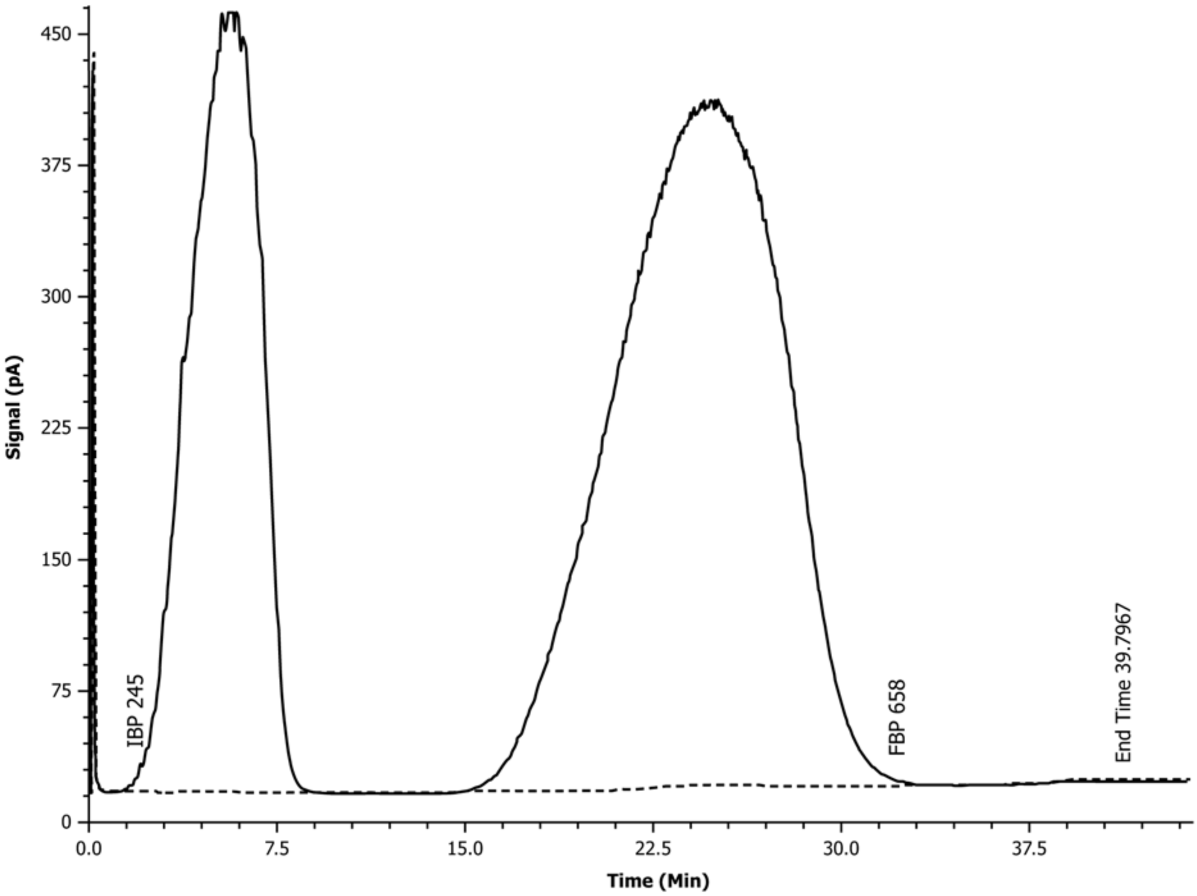

FIG. 4 Typical Chromatogram of Gravimetric Blend

chromatogram as shown in Fig. 7. If the sample baseline has an abrupt break and does not join the blank baseline, it is possible that the sample has not eluted completely from thee column and the sample is considered outside of the scope of this method.

11.6.1 Identify the start of the area of interest by selecting a point on the baseline where the blank and the sample baselines are merged. This is taken before the start of the sample and after the end of the solvent (Fig. 8).

11.6.2 Identify the end of the area of interest by selecting a point on the baseline where the blank and the sample baselines are merged. This is taken after the end of the sample and at or before the end of run (Fig. 8).

11.6.3 The start of the sample is determined by the software as given in A1.4.

11.6.4 The end of the sample is determined by the software as given in A1.5.

12. Calculation

12.1 The following calculations are performed. This section is intended as guide for understanding sequence of the calculations outlined in the Annex A1. The detailed steps are given in Annex A1.

12.1.1 Zero the sample signal after performing average and standard deviation of slices.

12.1.2 Zero the blank signal after performing average and standard deviation of slices.

12.1.3 Subtract blank from sample. Set negative slices to zero.

12.1.4 Calculate total chromatogram area.

12.1.5 Determine start of sample elution time.

12.1.6 Determine the end of sample elution time.

12.1.7 Calculated total corrected sample area.

12.1.8 Normalize to area percent.

12.1.9 Find retention time corresponding to percent off.

12.1.10 Convert retention time to boiling points.

12.1.11 Report the results.

13. Report

13.1 This section is intended to outline the required reports to be carried out. The actual calculations are detailed in Annex A1.

13.1.1 Report the 0.5 % (m/m) point as the initial boiling point and the 99.5 % (m/m) point as the final boiling point,

13.1.2 Report all temperatures to the nearest 1 ºC (1 °F),

13.1.3 Report all percentages to the nearest 1 % (m/m),

13.1.4 Report intermediate percentages as required at intervals of not less than 1 % (m/m).

13.2 The test report shall contain at least the following information:

13.2.1 A reference to this test method,

13.2.2 The type and complete identification of the material tested,

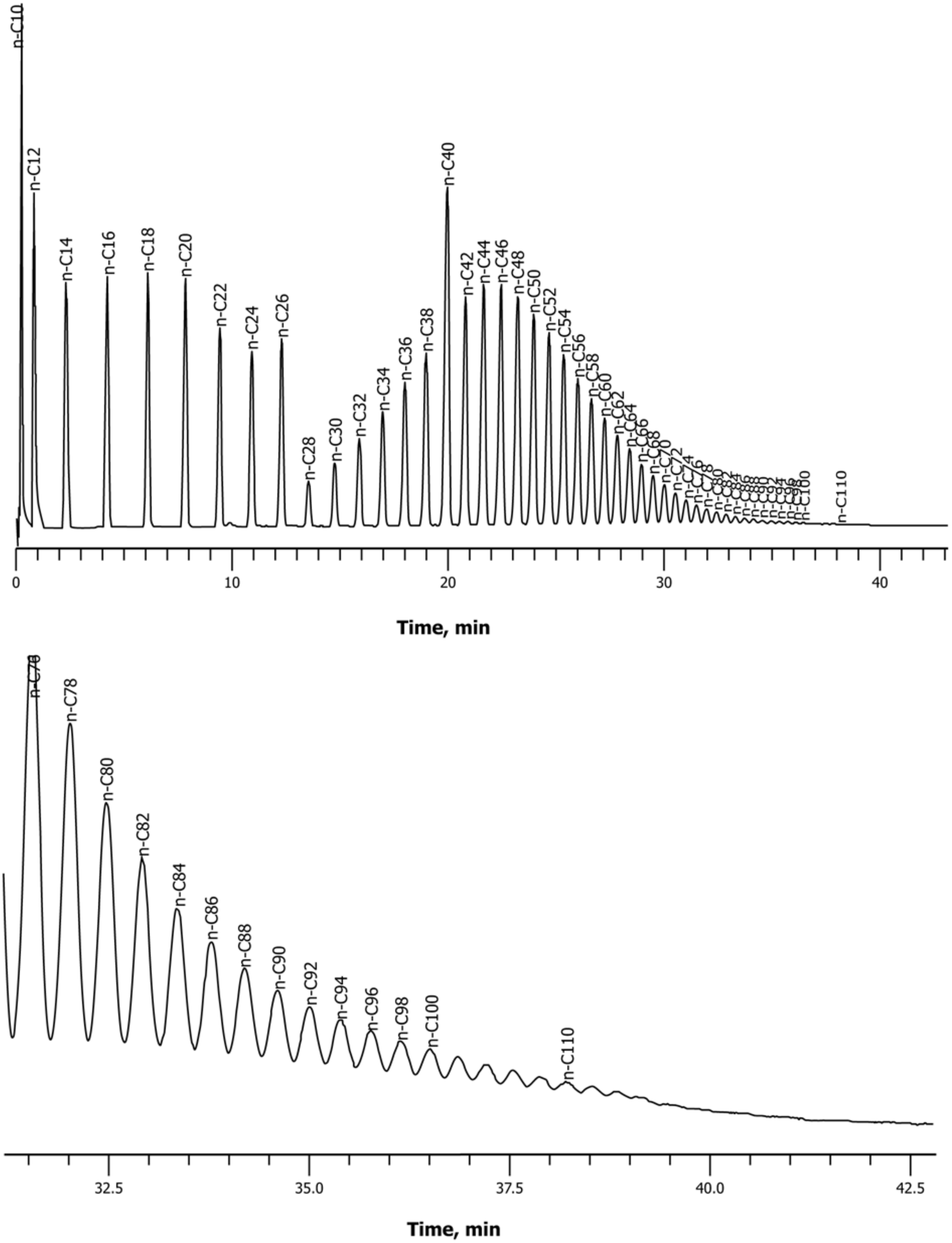

FIG. 5 Typical Chromatogram of Calibration Mix

13.2.3 The result of the test (see Section 12),

13.2.4 Any deviation, by agreement or otherwise, from the standard procedures specified, and

13.2.5 The date of the test.

TABLE 3 Repeatability and Reproducibility[A]

% Dist. M/M	Range of Results		Repeatability, r		Reproducibility, R	
	°C	°F	°C	°F	°C	°F
IBP	199–426	390–799	3	5.4	9	16
5 %	244–477	471–891	$0.09\sqrt{X}$	$0.09\sqrt{(F-32)/1.8}$	$0.20\sqrt{X}$	$0.20\sqrt{(F-32)/1.8}$
10 %	254–492	489–918	$0.08\sqrt{X}$	$0.08\sqrt{(F-32)/1.8}$	$0.18\sqrt{X}$	$0.18\sqrt{(F-32)/1.8}$
20 %	265–510	509–950	$0.07\sqrt{X}$	$0.07\sqrt{(F-32)/1.8}$	$0.22\sqrt{X}$	$0.22\sqrt{(F-32)/1.8}$
30–95 %	273–615	523–1139	1	1.8	5	9
FBP	389–655	732–1211	5	9	17	31

[A] Where X is the temperature obtained in °C and °F is the temperature in Fahrenheit.

14. Precision and Bias[6]

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results as recorded in RR:D02-1691 is as follows:

14.1.1 *Repeatability*—The differences 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 values presented in Table 3 in only one case in twenty.

14.1.2 *Reproducibility*—The differences between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values presented in Table 3 in only one case in twenty.

14.2 *Bias*—Because the boiling point distribution can be defined only in terms of a test method, no bias for the procedures in this test method as determined by gas chromatography has been determined.

15. Keywords

15.1 boiling range distribution; capillary gas chromatography; distillates; flame ionization detection; lubricating base oils; petroleum products; simulated distillation

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1691.

TABLE 4 Boiling Points of *n*-Paraffins[A,B]

Carbon No.	Boiling Point, °C	Boiling Point, °F
1	–162	–259
2	–89	–127
3	–42	–44
4	0	31
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	625
20	344	651
21	356	675
22	369	696
23	380	716
24	391	736
25	402	755
26	412	774
27	422	791
28	431	808
29	440	824
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
61	619	1146
62	622	1152
63	625	1157
64	629	1164
65	632	1170
66	635	1175
67	638	1180
68	641	1186
69	644	1191
70	647	1197
71	650	1202
72	653	1207
73	655	1211
74	658	1216
75	661	1222
76	664	1227
77	667	1233
78	670	1238
79	673	1243
80	675	1247
81	678	1252
82	681	1258
83	683	1261
84	686	1267
85	688	1270
86	691	1276
87	693	1279
88	695	1283
89	697	1287
90	700	1292
91	702	1296
92	704	1299
93	706	1303
94	708	1306
95	710	1310
96	712	1314
97	714	1317
98	716	1321
99	718	1324
100	720	1328
110[C]	735	1355

[A] API Project 44, October 31, 1972 is believed to have provided the original normal paraffin boiling point data that are listed in this table. 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. The values listed here as well as in Test Method D6352 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] Test Method D6352 has traditionally used *n*-paraffin boiling points rounded to the nearest whole degree for calibration. The boiling points listed in this table 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 209 °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.

[C] The boiling point of carbon 110 has been obtained by graphical extrapolation of the retention time versus boiling point curve shown in Fig. 3.

TABLE 5 Retention Time Data Corresponding to Fig. 5

Component	Time	BP (°C)	Skewness
n-C10	0.227	174.1	1.077
n-C12	0.811	216.3	0.703
n-C14	2.301	253.9	0.788
n-C16	4.218	287.2	1.474
n-C18	6.098	316.1	1.471
n-C20	7.842	343.9	1.473
n-C22	9.437	368.3	1.353
n-C24	10.919	391.1	1.347
n-C26	12.306	412.2	1.447
n-C28	13.553	431.1	1.116
n-C30	14.763	449.7	1.134
n-C32	15.906	466.1	1.152
n-C34	16.991	481.1	1.248
n-C36	18.019	496.1	1.322
n-C38	19.003	508.9	1.472
n-C40	19.987	522.2	1.672
n-C42	20.833	533.9	1.748
n-C44	21.675	545	1.683
n-C46	22.489	556.1	1.819
n-C48	23.257	566.1	1.71
n-C50	23.997	575	1.692
n-C52	24.706	583.9	1.623
n-C54	25.383	592.2	1.481
n-C56	26.043	600	1.506
n-C58	26.668	607.8	1.324
n-C60	27.274	615	1.235
n-C62	27.872	622.2	1.322
n-C64	28.441	628.9	1.251
n-C66	28.993	635	1.195
n-C68	29.527	641.1	1.133
n-C70	30.058	647.2	1.213
n-C72	30.558	652.8	1.107
n-C74	31.054	657.8	1.097
n-C76	31.538	663.9	1.115
n-C78	32.009	670	1.089
n-C80	32.456	675	0.942
n-C82	32.91	681.1	0.995
n-C84	33.343	686.1	0.922
n-C86	33.775	691.1	1.005
n-C88	34.188	695	0.909
n-C90	34.606	700	1.005
n-C92	35.003	703.9	1.011
n-C94	35.377	707.8	0.775
n-C96	35.767	712.2	0.945
n-C98	36.142	716.1	0.972
n-C100	36.507	720	0.983
n-C110	38.202	735	0.774

TABLE 6 Typical Values Obtained for Reference Oil 5010

% Off	BP (°C)	QC (°C)	(–) Diff	Limit
IBP	429.2	427.2	1.9	8.9
5	477.4	477.2	0.2	2.8
10	492.8	492.2	0.6	2.8
15	502.4	502.2	0.1	2.8
20	510.1	510	0.1	3.3
25	517.3	517.2	0.1	3.3
30	524.1	523.9	0.2	3.9
35	530.5	530.6	0	3.9
40	536.6	536.7	–0.1	4.4
45	542.4	542.2	0.2	4.4
50	548.2	548.3	–0.1	4.4
55	554.2	554.4	–0.2	4.4
60	560.1	560	0.1	4.4
65	566.1	566.1	0	4.4
70	571.8	572.2	–0.4	4.4
75	578	578.3	–0.3	5
80	585	585.6	–0.6	4.4
85	592.7	592.8	–0.1	3.9
90	602	602.2	–0.2	4.4
95	615.8	615.6	0.3	3.9
FBP	656.7	656.1	0.6	17.8

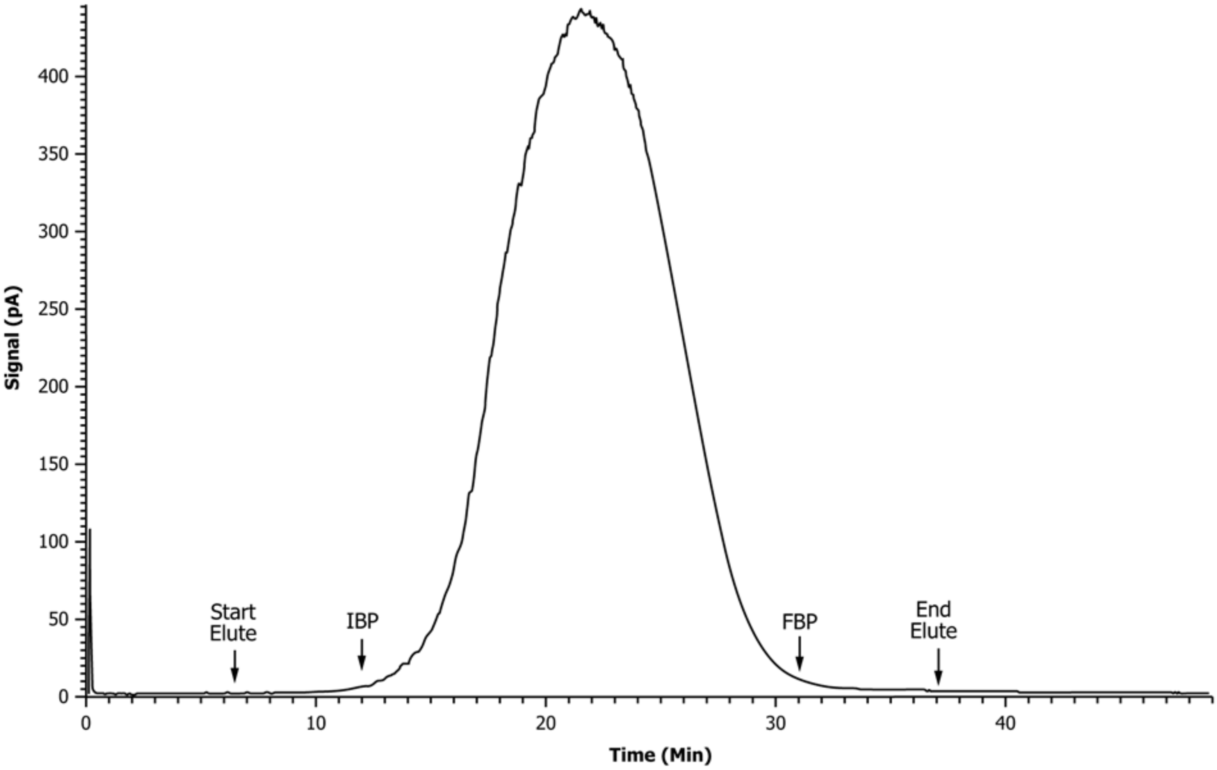

FIG. 6 Typical Chromatogram of Reference Oil 5010

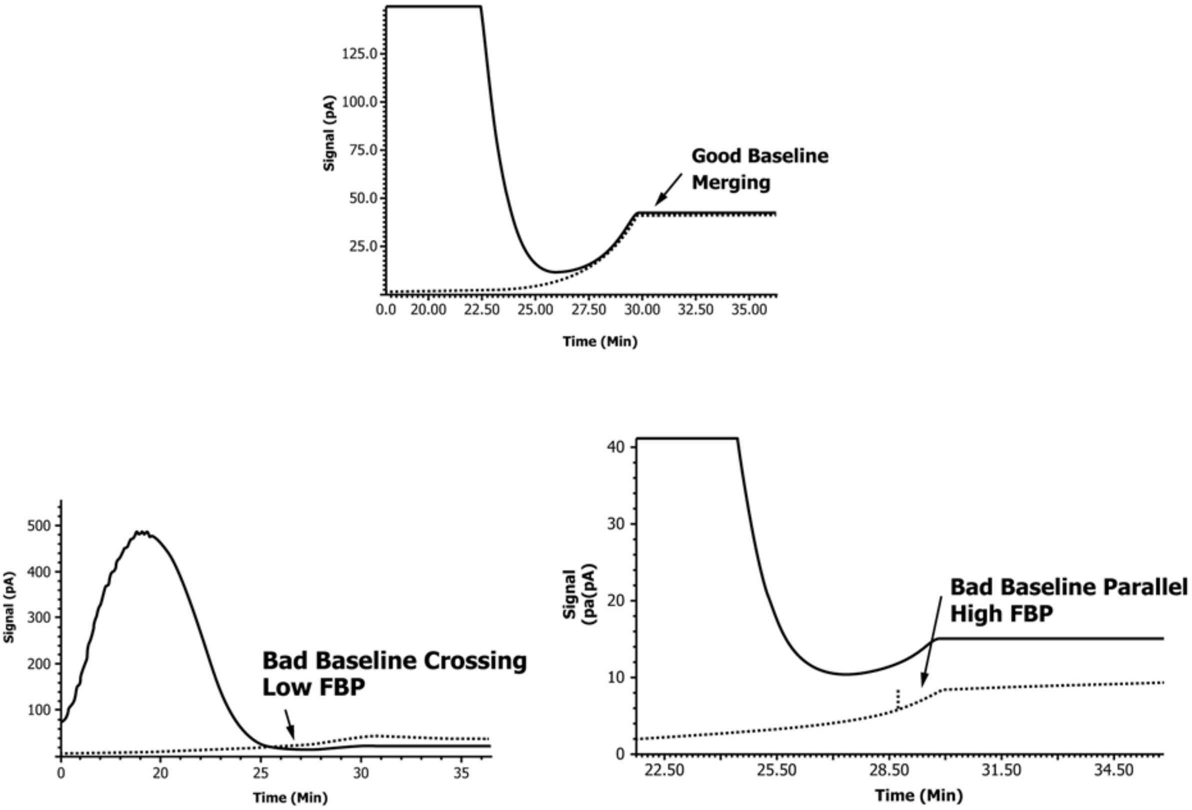

FIG. 7 Correct and Incorrect Positioning of Baseline Signal with Respect to Sample Signal

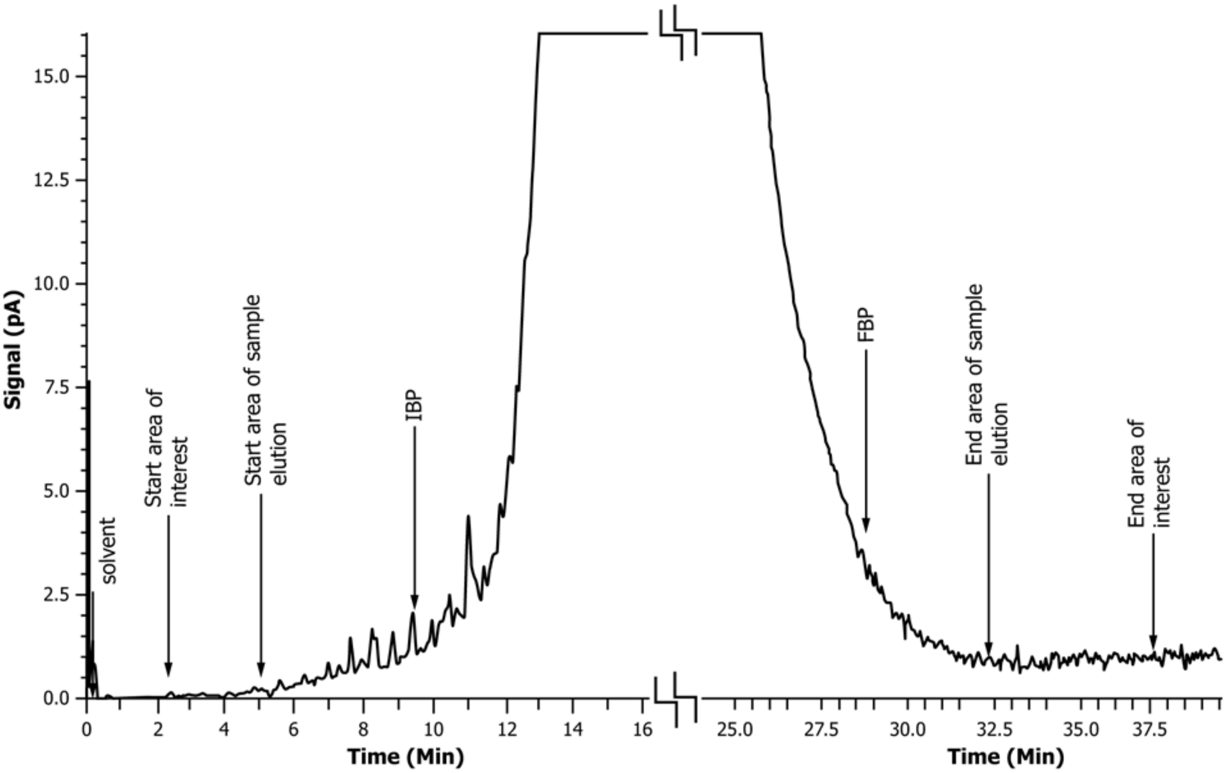

FIG. 8 Regions of the Enlarged Chromatogram to be Considered in the Developing of the Algorithm

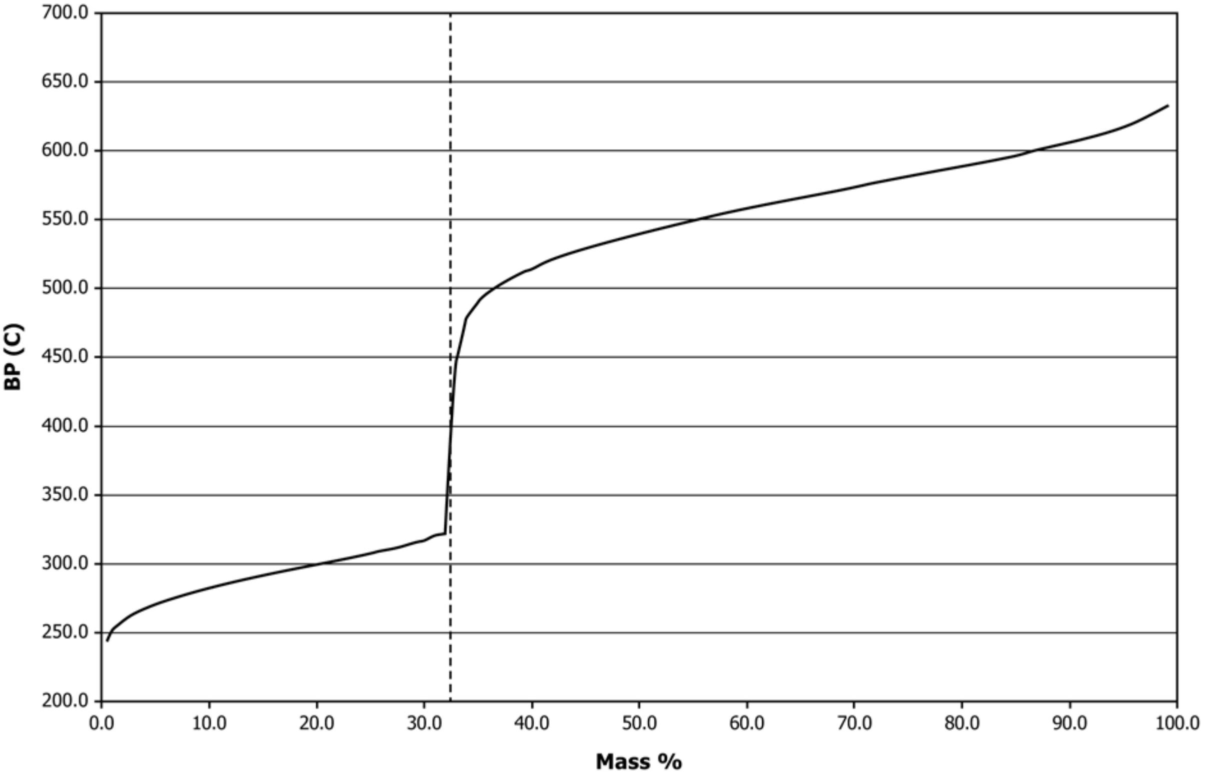

FIG. 9 Boiling Point Curve of the Gravimetric Blend

ANNEXES

(Mandatory Information)

A1. CALCULATIONS

A1.1 Acquisition Rate Requirements

A1.1.1 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.

A1.1.2 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. Ensure that the smallest number of slices is 5 or greater.

A1.1.3 Verify that the slice width used to acquire the sample chromatogram is the same used to acquire the blank run chromatogram.

A1.2 *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.

A1.2.1 *Sample Offset:*

A1.2.1.1 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.

A1.2.1.2 Subtract the average slice offset from all the slices of the sample chromatogram. Set negative slices to zero. This will zero the chromatogram.

A1.2.2 *Blank Offset:*

NOTE A1.1—If you are using electronic baseline compensation proceed to A1.4. It is recommended that a true baseline or blank be acquired in the slice mode of the Data Acquisition System. This blank is to be used for subtracting slice by slice from the signal.

A1.2.2.1 Repeat A1.2.1 using the accumulated blank run slice table.

A1.3 *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.

A1.4 Determine the Start of Sample Elution Time

A1.4.1 *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.

A1.4.2 *Calculate the Rate of Change between each Two Consecutive Area Slices*—Begin at the slice set in A1.4.1 and work forward. The rate of change is obtained by subtraction 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 A1.4.1) is defined as the start of the 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 1 s may be required.

A1.5 Determine the End of Sample Elution Time

A1.5.1 *Calculate the Rate of Change between each Two Consecutive Area Slices*—Begin at the end of run and work backward. 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 A1.4.1) 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 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.

A1.6 *Calculate 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.

A1.7 *Normalize to Area Percent*—Divide each slice in the sample chromatogram table by the total area (see A1.6) and multiply it by 100.

A1.8 Calculate the Boiling Point Distribution Table

A1.8.1 *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 A1.9.1) 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 A1.9.2).

A1.8.2 *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 A1.9.1) 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 A1.9.2).

A1.8.3 *Intermediate Boiling Point*—For each point between 1 % and 99 %, find the time where the accumulative sum is equal to or greater than the area percent being analyzed. As in A1.8.1 and A1.8.2, use interpolation when the accumulated sum exceeds the area percent to be estimated (refer to the algorithm in A1.9.1). Use the calibration table to assign the boiling point.

A1.9 Calculation Algorithms

A1.9.1 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 %.

A1.9.1.1 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.

A1.9.1.2 Calculate the fraction of the slice required to produce the exact X percent of the total area:

$$A_x = \frac{X - A_c}{A_{c+1} - A_c} \qquad (A1.1)$$

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

A1.9.1.3 Calculate the time required to generate the fraction of area, A_x:

$$T_f = A_x \times W \qquad (A1.2)$$

where:

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

A1.9.1.4 Record the exact time where the accumulative area is equal to the X percent of the total area:

$$T_t = T_s + T_f \qquad (A1.3)$$

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.

A1.9.2 Interpolate to determine the exact boiling point given the retention time corresponding to the cumulative slice area.

A1.9.2.1 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.)

A1.9.2.2 If the interpolation time is equal to the retention time of the standard, record the corresponding boiling point.

A1.9.2.3 If the retention time is not equal to a retention time of the standards, interpolate the boiling point temperature as follows:

A1.9.2.4 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:

$$BP_x = m_1 \times (RT_x - RT_1) + BP_1 \qquad (A1.4)$$

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.

A1.9.2.5 If the interpolation time is between two retention times in the calibration table, then interpolate using the upper and lower standard components:

$$BP_x = m_u \times (RT_x - RT_1) + BP_1 \qquad (A1.5)$$

where:

m_u = $(BP_u - BP_1) / (RT_2 - RT_1)$
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_2 = retention time of the upper bound component in the calibration table, and
BP_2 = boiling point of the upper bound component in the calibration table.

A1.9.2.6 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:

$$BP_x = m_n \times (RT_x - RT_{n-1}) + BP_{n-1} \qquad (A1.6)$$

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.

A1.10 Report

A1.10.1 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. Print the Boiling Point Distribution either using spreadsheet or commercial simulated distillation software.

A2. BOILING POINTS OF NORMAL ALKANES

A2.1 The boiling points of normal alkanes used for construction of the calibration curve are given in Table 4.

A3. SYSTEM PERFORMANCE CHECK

A3.1 Frequency

A3.1.1 Carry out a run on the calibration mix (7.8), using identical conditions, and injection volumes to those used for the sample analysis at the start of an analysis. Periodically inject this mix to verify the shift in retention time. Also this calibration mix shall be injected if the analytical system and conditions have been altered in any way since the last performance check was carried out or whenever the results obtained for the reference oil 5010 fall outside the permitted limits.

A3.1.2 A typical chromatogram of the calibration mix is shown in Fig. 5.

NOTE A3.1—This procedure may be carried out as part of the boiling range calibration (see Section 10).

A3.2 Column Resolution

A3.2.1 Determine the column resolution, *R*, using the C_{50} and C_{52} peaks and the following equation:

$$R = \frac{2(t_2 - t_1)}{1.699\,(W_1 + W_2)} \quad (A3.1)$$

where:

t_1 = the retention time, in seconds, for the C_{50} peak,
t_2 = the retention time, in seconds, for the C_{52} peak,
W_1 = the width, in seconds, at half-height of C_{50} peak, and
W_2 = the width, in seconds, at half-height of C_{52} peak.

A3.2.2 Resolution as determined above shall be at least 2, but no greater than 4.

A3.3 Detector Response

A3.3.1 Use a binary gravimetric blend distillate to determine the detector response as well as to determine the quality of the baseline. Since the most critical area of the chromatogram is where column bleeding occurs, the binary blend is also used as a recovery of the baseline test. The binary blend shall have the following characteristics:

A3.3.2 It shall consists of two fractions: The first fraction 1 shall have an IBP 240.5 °C and a FBP 362 °C. The second fraction 2 shall have an IBP of 459 °C and a FBP of 652 °C.

A3.3.3 Ensure that the two fractions have similar height response and follow the criteria of not exceeding linearity nor overloading the column.

A3.3.4 The ratio of the area's of the two distillates shall be constant and meet the following conditions:

A3.3.4.1 The lower boiling distillate shall not interfere with the solvent.

A3.3.4.2 There shall be a baseline between distillates.

A3.3.4.3 The higher boiling point distillate shall elute totally and as close to the end of the temperature program as possible.

A3.3.4.4 The two fractions shall preferably contain no aromatics so that the detector response is due to saturates only.

A3.3.5 A typical chromatogram of the gravimetric blend with its superimposed baseline is shown in Fig. 4.

A3.3.6 With the example given this is best achieved with a mixture of fraction 1 of 32 %. The determined mass % is obtained from the distillation curve which presents a break at 32.4 % ± 0.6 % at 400 °C which corresponds to the midpoint of the distillation curve of the composite fractions as shown in Fig. 9.

A3.4 Peak Skewness

A3.4.1 Determine the skewness of the calibration mix peaks as the ratio *A/B* as shown in Fig. 3, for the C_{20} peak,

where:

A = the width of the leading part of the peak at 5 % of the peak height, and

B = the width of the following part of the peak at 5 % of the peak height.

A3.4.2 The ratio shall for all peaks be between 0.8 and 1.8.

A3.4.3 Verify that the skewness of all peaks does not exceed the limits. Typical skewness data for the calibration mix chromatogram of Fig. 5 is shown in Table 5.

APPENDIX

(Nonmandatory Information)

X1. OPERATING CONDITIONS FOR GAS CHROMATOGRAPH USING ALTERNATIVE CARRIER GASES

NOTE X1.1—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.)

X1.1 More often laboratories are looking for other carrier gases in their gas chromatographic analyses than helium for performance or cost reasons, or both. This appendix gives some operating guidelines for alternative carrier gases that can be used in the simulated distillation analysis as described in this test method.

X1.2 Typical alternative carrier gases to use are hydrogen or nitrogen. These gases should have a purity of at least 99.999 % (v/v). Any oxygen present is removed by a chemical resin filter. (**Warning—**Follow the safety instructions from the filter supplier.) Total impurities are not to exceed 10 mL/m^3.

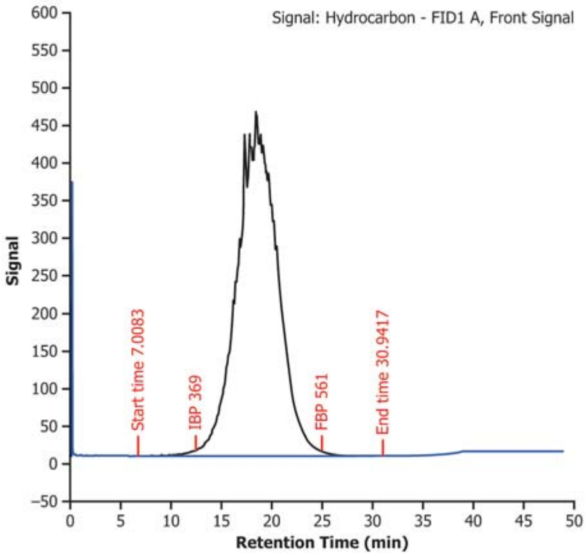

FIG. X1.2 Chromatogram of the Reference Gas Oil Utilizing N_2 as Carrier Gas

X1.3 The system configuration and temperature program as described in Table X1.1 are valid for all carrier gases. The deviations in operating conditions between the gases are given in Table X1.2.

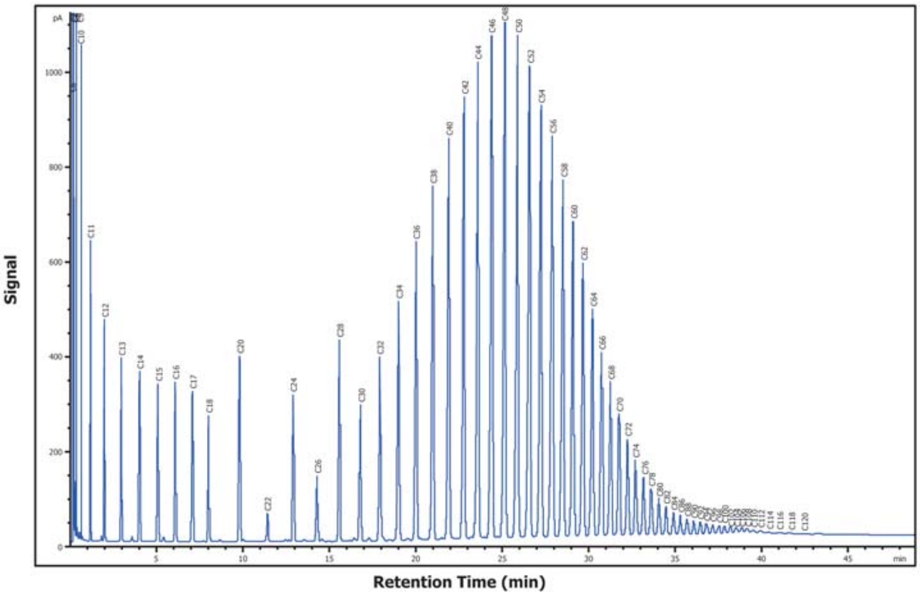

FIG. X1.1 Chromatogram from the Calibration Sample Utilizing H_2 as Carrier Gas

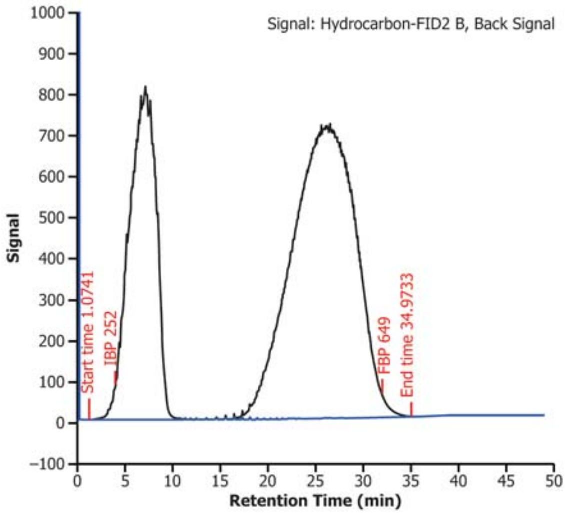

FIG. X1.3 Chromatogram of the Gravimetric Blend Utilizing H_2 as Carrier Gas

TABLE X1.1 Typical Operating Conditions for Gas Chromatograph using Hydrogen or Nitrogen as Carrier Gas

	Nitrogen	Hydrogen
Column flow (mL/min)	10	19
FID (Hydrogen)	35	15
FID (Air)	350	350
Make up (Nitrogen)	20	20

Column used: 5 m × 0.530 mm × 0.17 µm PDMS
Oven program: 40 °C to 430 °C at 10 °C ⁄min. Hold time 10 min.

TABLE X1.2 ASTM Reference Gas Oil Boiling Point distribution Values obtained with H2 (left) and with N2 (right) Carrier Gases

Reference Check					Reference Check				
Recovered Mass %	Target Values BP°C	dBP°C	Determined Values BP°C	dBP°C	Recovered Mass %	Target Values BP°C	dBP°C	Determined Values BP°C	dBP°C
IBP	366.0	6.0	368.5	2.5	IBP	366.0	6.0	369.2	3.2
10.0	421.5	3.0	423.6	2.1	10.0	423.0	3.0	424.1	1.1
20.0	436.0	3.0	437.3	1.3	20.0	436.0	3.0	437.4	1.4
30.0	445.5	3.0	446.6	1.1	30.0	445.5	3.0	446.3	0.8
40.0	454.0	3.0	454.8	0.8	40.0	454.0	3.0	454.3	0.3
50.0	462.0	4.0	462.3	0.3	50.0	462.0	4.0	461.7	–0.3
60.0	469.5	4.0	469.7	0.2	60.0	469.5	4.0	469.1	–0.4
70.0	477.5	4.0	477.4	–0.1	70.0	477.5	4.0	476.9	–0.6
80.0	486.0	4.0	486.2	0.2	80.0	486.0	4.0	485.9	–0.1
90.0	498.0	4.0	498.2	0.2	90.0	498.0	4.0	498.5	0.5
FBP	544.5	8.5	541.8	–2.7	FBP	544.5	8.5	551.3	6.8

SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D7500 – 14) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Revised Note X1.1.

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D7500 – 12) that may impact the use of this standard. (Approved Oct. 1, 2014.)

(1) Added Appendix X1.

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