Designation: D5307 – 97 (Reapproved 2007)

Standard Test Method for Determination of Boiling Range Distribution of Crude Petroleum by Gas Chromatography[1]

This standard is issued under the fixed designation D5307; 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 water-free crude petroleum through 538°C (1000°F). Material boiling above 538°C is reported as residue. This test method is applicable to whole crude samples, that can be solubilized in a solvent to permit sampling by means of a microsyringe.

1.2 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are for information only.

1.3 *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.* Specific precautionary statements are given in 7.2, 7.5, 7.6, 7.7, and 7.9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *area slice*, *n*—the area, resulting from the integration of the chromatographic detector signal, within a specified retention time interval.

3.1.1.1 *Discussion*—In area slice mode (see 6.2.2), peak detection parameters are bypassed and the detector signal integral is recorded as area slices of consecutive, fixed duration time intervals.

3.1.2 *corrected area slice*, *n*—an area slice corrected for baseline drift, by subtraction of the exactly corresponding area slice in a previously recorded blank (nonsample) analysis; correction for signal offset may also be required.

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

3.1.4 *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 theoretical total area is obtained.

3.1.5 *residue, RES n*—the amount of sample boiling above 538°C (1000°F).

3.1.6 *theoretical total area, T n*—the area that would have been obtained if the entire sample had been eluted from the column.

3.1.6.1 *Discussion*—This is determined in 12.3.

3.2 *Abbreviations:Abbreviations:*

3.2.1 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}; isotetradecane = i-C_{14}).

4. Summary of Test Method

4.1 The crude oil sample is diluted with carbon disulfide, and the resulting solution is injected into a gas chromatographic column that separates hydrocarbons in boiling point order. The column temperature is raised at a reproducible, linear rate, and the area under the chromatogram is recorded throughout the run. Boiling points are assigned to the time axis by comparison to a calibration curve obtained under the same chromatographic conditions by running a mixture of n-paraffins of known boiling point through a temperature of 538°C (1000°F). The amount of sample boiling above 538°C is estimated by means of a second analysis of the crude oil to which an internal standard has been added. From these data, the boiling range distribution of the water-free sample is calculated.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

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5. Significance and Use

5.1 The determination of the boiling range distribution is an essential requirement in crude oil assay. This information can be used to estimate refinery yields and, along with other information, to evaluate the economics of using one particular crude as opposed to another.

5.2 Results obtained by this test method are equivalent to those obtained from Test Method D2892. (See Appendix X1.)

5.3 This test method is faster than Test Method D2892 and can be used when only small volumes of samples are available. Also, this test method gives results up to 538°C while Test Method D2892 is limited to 400°C.

6. Apparatus

6.1 *Gas Chromatograph*—Any gas chromatograph may be used that has the capabilities described below and meets the performance requirements in Section 10.

6.1.1 *Detector*—This test method is limited to the use of the flame ionization detector (FID). The detector must be capable of operating continuously at a temperature equal to or greater than the maximum column temperature employed, and it must be connected to the column so as to avoid cold spots.

6.1.2 *Column Temperature Programmer*—The chromatograph must be capable of reproducible, linear programmed temperature operation over a range sufficient to establish a retention time of at least 1 min for the IBP and to elute compounds with boiling points of 538°C (1000°F) before the end of the temperature ramp.

6.1.3 *Cryogenic Column Oven*—If the IBP of the crude oil is below 90°C (194°F), an initial column temperature below ambient will be required. This necessitates a cryogenic cooling option on the gas chromatograph. Typical initial column temperatures are listed in Table 1.

6.1.4 *Sample Inlet System*—Either of the following two types of sample inlet systems may be used.

6.1.4.1 *Flash Vaporization*—A vaporizing sample inlet system must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed. The sample inlet system also must be connected to the chromatographic column so as to avoid any cold spots.

6.1.4.2 *On-Column*—Capable of introducing a liquid sample directly onto the head of the column. Means must be provided for programming the entire column, including the point of sample introduction, up to the maximum column temperature employed.

6.1.5 *Flow Controller*—The chromatograph must be equipped with a flow controller capable of maintaining carrier gas flow constant to ± 1 % over the full operating temperature range of the column. The inlet pressure of the carrier gas, supplied to the chromatograph, must be sufficiently high to compensate for the increase of backpressure in the column as the temperature is programmed upward. An inlet pressure of 550 kPa gage (80 psig) has been found satisfactory with the columns described in Table 1.

6.2 *Data Retrieval System*:

6.2.1 *Recorder*—A 0–1 mV range recording potentiometer or equivalent, with a full-scale response time of 2 s or less may be used for graphic presentation of the FID signal.

6.2.2 *Integrator*—Electronic integrator or computer-based chromatography data system must be used for detector signal integration and accumulation. The integrator/computer system must have normal chromatographic software for measuring retention time and areas of eluting peaks (peak detection mode). In addition, the system must be capable of converting the continuously integrated detector signal into area slices representing contiguous fixed duration time intervals (area slice mode). The recommended time interval is 1 s. No time interval shall be greater than 12 s. The system must be capable of subtracting the area slice of a blank run from the corresponding area slice of a sample run. Alternatively, the baseline chromatogram can be subtracted from the sample chromatogram and the net resulting chromatogram can be processed in the slice mode. A computer program that performs the slice calculation as a post-run calculation is also used.

6.3 *Column*—Any gas chromatographic column that provides separation in order of boiling points and meets the performance requirements of Section 10 can be used. Columns and conditions, which have been used successfully, are shown in Table 1.

6.4 *Microsyringe*—A 5 or 10 µL syringe is used for sample introduction. The use of an automated liquid sampling device is highly recommended.

TABLE 1 Typical Operating Conditions

	1	2	3
Column length, mm (in.)	457 (18)	610 (24)	457 (18)
Column diameter, mm (in.)	3.17 (1⁄8)	3.17 (1⁄8)	3.17 (1⁄8)
Liquid phase	10 % UCW-982	3 % OV-1	10 % SE-30
Support material	Chromosorb P[A]-AW	Chromosorb W[A]-HP	Chromosorb P[A]-AW
Column temperature initial value,° C	−30	−30	−40
Column temperature final value,° C	380	350	360
Programming rate, °C/min	10	10	10
Carrier gas type	N_2	He	N_2
Carrier gas flow, mL/min	25	20	28
Detector temperature, °C	400	380	400
Injection port temperature, °C	380	375	400

[A]See Footnote 5.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.2 *Air*—Zero grade (hydrocarbon free) for use with the FID. (**Warning**—Air is a compressed gas under high pressure and supports combustion.)

7.3 *Calcium Chloride, Anhydrous ($CaCl_2$).*

7.4 *Calibration Mixture*—A mixture of *n*-paraffins dissolved in carbon disulfide (**Warning**—see 7.5) covering the boiling range of the sample through 538°C (1000°F). At least one compound in the mixture must have a boiling point equal to or lower than the IBP of the sample. Methane, ethane, propane, or butane can be added to the calibration mixture, if necessary, by injecting about 1 mL of the pure gaseous compound into a septum-capped, sealed vial containing the rest of the calibration mixture, using a gas syringe. If *n*-paraffin peaks can be unambiguously identified in the sample chromatogram, their retention times can be used for calibration.

7.5 *Carbon Disulfide (CS_2)*—Carbon disulfide (99 % minimum purity) is used as a viscosity reducing solvent because it is miscible with crude oils and has only a slight response with the FID. (**Warning**—Carbon disulfide is extremely volatile, flammable, and toxic.)

7.6 *Carrier Gas*—Nitrogen or helium of high purity that has been dried over molecular sieves or similar suitable drying agents. (**Warning**—Helium and nitrogen are compressed gases under high pressure.)

7.7 *Column Resolution Test Mixture*—A mixture of 1 % each of *n*-C_{16} and *n*-C_{18} paraffin in a suitable solvent, such as *n*-octane, for use in testing the column resolution. (**Warning**—*n*-Octane is flammable and harmful if inhaled.)

7.8 *Detector Response Test Mixture*—An accurately weighed mixture of approximately equal masses of at least six *n*-paraffins covering the carbon number range from 10 to 44. Dissolve one part of this mixture with approximately five parts of CS_2(or sufficient CS_2 to ensure a stable solution at room temperature).

7.9 *Hydrogen*—Hydrogen of high quality (hydrocarbon free) is used as fuel gas for the FID. (**Warning**—Hydrogen is an extremely flammable gas under high pressure.)

7.10 *Internal Standard*—A mixture of approximately equal amounts of four *n*-paraffins, *n*-C_{14} through *n*-C_{17}. Concentrations of the individual components need not be known but must be within the linear range of the detector/electronics system used.

7.11 *Liquid Phase*—A nonreactive, nonpolar liquid or gum of low volatility. Silicone gum rubbers are typically used. In general, liquid phase loadings of 3 to 10 % have been found most satisfactory.

7.12 *Solid Support*—A diatomaceous earth or equivalent nonreactive particulate material. Typical particle size ranges are 60/80 or 80/100 mesh.

8. Sampling

8.1 Obtain samples for analysis by this test method in accordance with instructions given in Practice D4057.

8.1.1 Ensure that samples are received in sealed containers and show no evidence of leakage.

9. Preparation of Apparatus

9.1 *Column Preparation*—Any satisfactory method used in the practice of the art, that will produce a column meeting the requirements of Section 10, may be used.

9.2 *Column Conditioning*—The column must be conditioned at the maximum operating temperature to reduce baseline shifts due to bleeding of the column substrate. The column can be conditioned rapidly and effectively using the following procedure:

9.2.1 Connect the column to the inlet system but leave the detector end free.

9.2.2 Purge the column at ambient temperature with carrier gas.

9.2.3 Turn off the carrier gas and allow the column to depressurize completely.

9.2.4 Seal off the open end of the column with an appropriate fitting.

9.2.5 Raise the column to the maximum operating temperature and hold at this temperature 4 to 6 h, with no flow through the column.

9.2.6 Cool the column to ambient temperature.

9.2.7 Remove the cap from the column and connect the column to the detector. Re-establish carrier flow.

9.2.8 Program the column temperature to the maximum several times with normal carrier gas flow rate.

9.3 An alternate method of column conditioning, that has been found effective with columns with an initial loading of 5 % liquid phase, consists of purging the column (disconnected from the detector) with normal carrier gas flow rate for 12 to 16 h, while holding the column at the maximum operating temperature.

9.4 *Chromatograph*—Place the chromatograph in service in accordance with the manufacturer's instructions. Typical operating conditions are shown in Table 1.

9.4.1 Excessively low initial column temperature must be avoided to ensure that the column phase functions as gas-liquid chromatographic column. Consult the stationary phase manufacturer's literature for minimum operating temperature. The initial temperature of the column should be only low enough to obtain a calibration curve meeting the specifications under 6.1.3.

9.4.2 Silica from combustion of column material deposits on the FID parts. This deposit must be removed regularly, by brushing, because it changes response characteristics of the detector.

9.4.3 Silica deposits also can plug the end of the flame jet. This problem can be alleviated greatly by utilizing a flame jet with an inside diameter of at least 0.76 mm (0.030 in.)

10. System Performance

10.1 *Resolution*—Analyze an aliquot of the column resolution test mixture (see 7.7) utilizing identical conditions as used in the analysis of samples. The resolution of *n*-C_{16} and *n*-C_{18}*n*-paraffin peaks must be between three and ten when calculated in accordance with the following equation (refer to Fig. 1):

$$R = [2(t2 - t1)]/[1.699(Y2 + Y1)] \quad (1)$$

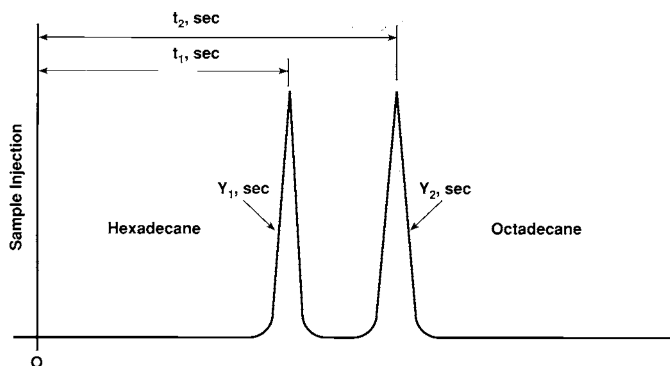

FIG. 1 Column Resolution Parameters

where:

R = resolution,
$t1$ = time for the n-C_{16} peak apex, in seconds,
$t2$ = time for the n-C_{18} peak apex, in seconds,
$Y1$ = peak width, at half height, of n-C_{16}, in seconds, and
$Y2$ = peak width, at half height, of n-C_{18}, in seconds.

10.2 *Retention Time Repeatability*—The system must be sufficiently repeatable, when testing with the calibration mixture, to obtain retention time repeatability (maximum difference between duplicate runs) of 6 s or less for each calibration peak.

10.3 *System Performance Check*—Analyze the detector response test mixture (see 7.8) utilizing identical conditions as used in the analysis of samples. Calculate response factors relative to n-decane as follows:

$$F_n = (C_n/A_n)/(C_{10}/A_{10}) \quad (2)$$

where:

A_n = area of that n-paraffin peak,
A_{10} = area of n-decane peak,
C_n = concentration of that n-paraffin in the mixture,
C_{10} = concentration of n-decane in the mixture, and
F_n = response factor relative to n-decane.

10.3.1 The response factor (F_n) of each n-paraffin must not deviate from unity by more than 10 %.

10.3.2 With some chromatographs, response factors for higher boiling n-paraffins (n-C_{20} to n-C_{44}) have been observed to change after several crude oil samples have been analyzed. Check the stability of the system by repeating the performance test after analyzing ten samples. If the system still meets the performance specified (see 10.3.1), it is not necessary to repeat this check after subsequent analyses. However, it is good practice to repeat the performance test if detector components are changed.

11. Procedure

11.1 *Baseline Compensation Analysis*—To compensate for baseline drift and signal offset, subtract an area slice profile of a blank run from the sample run to obtain corrected area slices. This profile is obtained as follows:

11.1.1 After conditions have been set to meet performance requirements, program the column oven temperature upward to the maximum temperature to be used and hold for at least ten minutes.

11.1.2 Following a rigorously standardized schedule, cool the column to the selected starting temperature, and allow it to equilibrate at this temperature for at least 3 min. At the exact time set by the schedule, without injecting a sample, start the column temperature program.

11.1.3 Acquire the data in area slice mode (see 6.2.2), recording the area slices for each time interval from the start of the run until the end of the run. It is essential that all measurements be on the same time basis for the blank and sample runs.

11.1.4 Perform a blank analysis at least once each day analyses are performed.

NOTE 1—A completely satisfactory baseline is difficult to obtain when compensation for column bleed is attempted with matched dual columns and detectors. In actual practice, the best compensation can be obtained by directly subtracting the area profile of the blank run derived from a single column.

NOTE 2—Some commercially available gas chromatographs have the capability to make baseline corrections (from a stored blank analysis) directly on the detector signal. Further correction of area slices may not be required with such systems. 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.

11.2 *Retention Time Versus Boiling Point Calibration*:

11.2.1 Using the same conditions as for the blank run, and following the same rigorously standardized schedule (see 11.1), inject an appropriate aliquot of the calibration mixture (see 7.4) into the chromatograph. Record the data in such a manner that retention times and areas for each component are obtained (peak detection mode).

11.2.1.1 The volume of the calibration mixture injected must be selected to avoid distortion of any component peak shapes caused by overloading the sample capacity of the column. Distorted peaks will result in displacement of peak apexes (that is, erroneous retention times) and hence errors in boiling point determination. The column liquid phase loading has a direct bearing on acceptable sample size.

11.2.2 Plot the retention time of each peak versus the corresponding boiling point for that component, as shown in Fig. 2. Boiling points of n-paraffins are listed in Table 2. Tabulate these same data and save for later calculations.

11.2.3 The calibration curve should be essentially a linear plot of boiling point versus retention time. Since it is not practical to operate the column so as to completely eliminate curvature at the lower end of the curve, the calibration mixture must contain at least one n-paraffin with a boiling point equal to or lower than the IBP of the sample. Extrapolation of the curve at the upper end (to 538°C) is more accurate provided extrapolation is not made outside the temperature-programmed portion of the run. However, for best accuracy, calibration points should bracket the boiling range to be measured at both low and high ends. If normal paraffins can be unambiguously identified in the sample, these retention times may be used for calibration.

11.2.4 Perform a boiling point-retention time calibration at least once each day analyses are performed.

11.3 *Sample Preparation*:

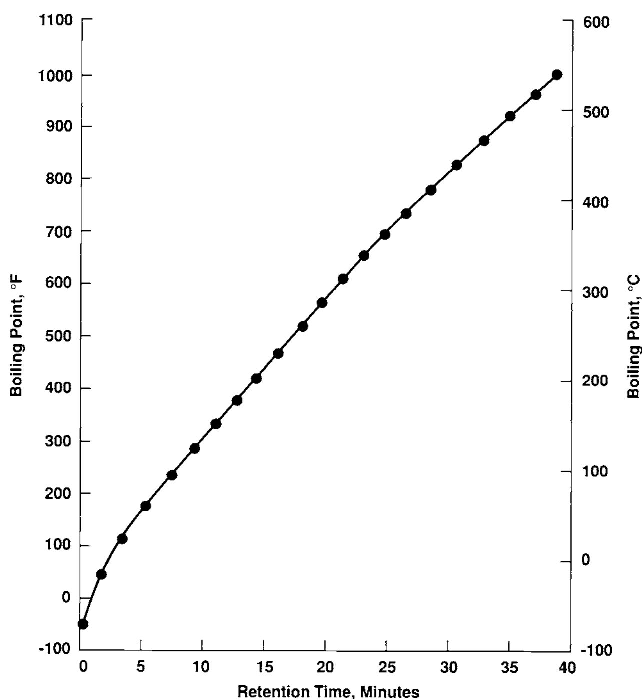

FIG. 2 Typical Calibration Curve

TABLE 2 Boiling Points of Normal Paraffins[A]

Carbon Number	BP, °C	BP, °F	Carbon Number	BP, °C	BP, °F
1	−162	−259	23	380	716
2	−89	−128	24	391	736
3	−42	−44	25	402	756
4	0	32	26	412	774
5	36	97	27	422	792
6	69	156	28	431	808
7	98	208	29	440	825
8	126	259	30	449	840
9	151	304			
10	174	345	31	458	856
			32	466	871
11	196	385	33	474	885
12	216	421	34	481	898
13	235	455	35	489	912
14	254	489	36	496	925
15	271	520	37	503	937
16	287	549	38	509	948
17	302	576	39	516	961
18	316	601	40	522	972
19	330	626			
20	344	651	41	528	982
			42	534	993
21	356	674	43	540	1004
22	369	695	44	545	1013

[A]See Footnote 7.

11.3.1 Store very light samples to between 0 and 5°C. Allow the unopened sample to remain within this temperature range for at least 4 h (preferably overnight) before opening.

11.3.2 Shake or stir the sample to ensure homogeneity and pour out a small portion (approximately 100 mL) for subsequent weighing and analysis.

11.3.3 Heavy, viscous crude may require warming as well as stirring to ensure homogeneity.

11.3.4 Since water is not measured by the FID, a portion of the sample must be dried before the sample can be weighed. Add 2 to 3 g of drying agent, such as anhydrous calcium chloride, to a 50-mL vial and fill the vial about half full with sample. Cap the vial tightly and shake the vial vigorously. Allow the mixture to stand several minutes to allow the drying agent to settle out. By means of a disposable pipette, remove the dried oil layer for sample weighing and analysis.

11.3.5 Weigh at least 10 g of dried sample to the nearest 0.1 mg into a 25-mL vial.

11.3.6 Add approximately 1 g of internal standard mixture into the same vial. Determine the weight to the nearest 0.1 mg.

11.3.7 Dilute the mixture with an approximately equal volume of carbon disulfide.

11.3.8 Cap the vial tightly and shake the mixture vigorously for 3 min, or until the mixture is solubilized completely. Use this solution for the crude oil plus internal standard analysis (see 11.4.1).

11.3.9 In a second vial, dissolve approximately the same amount of dried sample as 11.3.5 with an approximately equal volume of carbon disulfide. Use this solution for the separate crude oil without internal standard analysis (see 11.4.4).

11.4 *Sample Analysis*:

11.4.1 Using the exact conditions that were used in the blank and calibration runs (see 11.1 and 11.2), and following the rigorously defined schedule (see 11.1), inject 1 µL of the diluted crude oil plus internal standard mixture into the chromatograph. Record the area slices of each time interval through the end of the run.

11.4.2 Continue the run until the retention time equivalent to a boiling point of 538°C (1000°F) is reached. Stop recording area slices under the chromatogram at this point.

11.4.3 To remove as much as possible of the heavy components remaining on the column, continue heating the column until the FID signal returns to baseline. The column temperature may be increased to speed this process.

11.4.4 Cool the column to the starting temperature. Use identical conditions as used in 11.4.1. Inject 1 µL of the crude oil sample without internal standard (see 11.3.9). Record the area slices of each time interval through the end of the run.

11.4.5 The sample plus internal standard analysis (see 11.4.1) and the sample only analysis (see 11.4.4) may be made in either order.

12. Calculation

12.1 *Area Corrections*:

12.1.1 Obtain corrected area slices for both runs (see 11.4.1 and 11.4.4) by subtracting the corresponding area slice of the blank run profile (see 11.1) from each. (See Note 2.)

12.1.2 Sum the corrected area slices for both runs to obtain the cumulative corrected area at the end of each time interval during the run.

12.2 *Theoretical Total Area (Refer to Fig. 3)*:

12.2.1 Based on retention times from the calibration chromatogram (see 11.2.1), select a retention time that is 5 % less than the retention time of n-C_{14}, and another that is 5 % greater than the retention time of n-C_{17}. These times define a segment of the chromatogram that includes the internal standard peaks. Record the total area within this segment from the chromatogram of the crude oil plus internal standard mixture (see 11.4.1) (area *AIS* from Fig. 3(*a*)). Also record the total area of

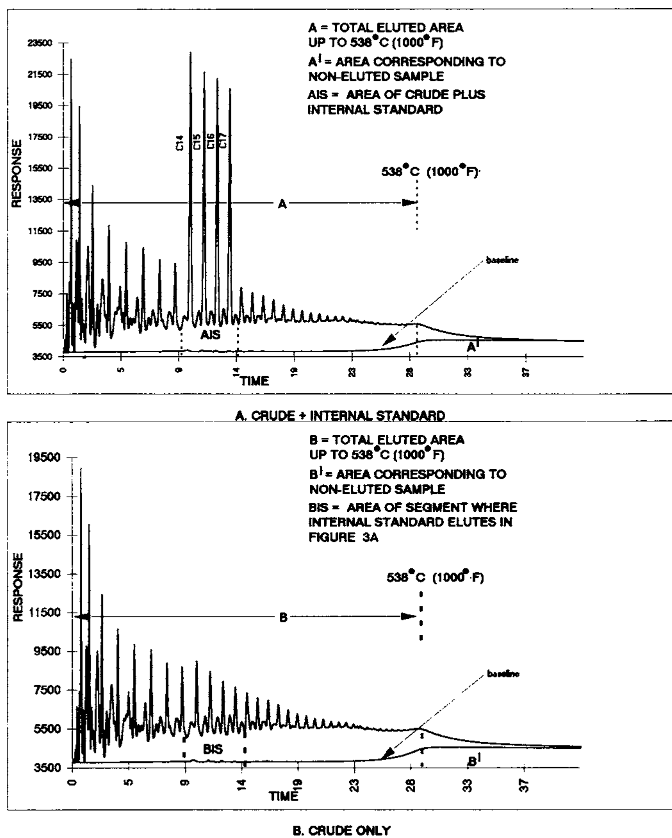

FIG. 3 Typical Chromatograms

the same segment from the chromatogram obtained from the crude oil only chromatogram (see 11.4.4) (area *BIS* from Fig. 3(*b*)).

12.2.2 Record the total area of both chromatograms through the retention time equivalent to a boiling point of 538°C (1000°F).

12.2.3 Calculate the mass fraction (W) of the internal standard in the mixture of sample plus internal standard as follows:

$$W = I/(S + I) \quad (3)$$

where:

I = mass of internal standard, g, and
S = mass of sample, g.

12.2.4 Calculate the ratio of areas (r) outside the internal standard segment and through the retention time equivalent to a boiling point of 538°C (1000°F) of the crude oil only chromatogram to the chromatogram from the mixture of internal standard plus crude as follows:

$$r = (B - BIS)/(A - AIS) \quad (4)$$

where:

A = total area through 538°C (1000°F) of the crude plus internal standard mixture chromatogram,
AIS = total area of the internal standard segment of the crude plus internal standard mixture chromatogram,
B = total area through 538°C (1000°F) of the crude oil only chromatogram, and
BIS = total area of the internal standard segment of the crude oil only chromatogram.

12.2.5 Calculate the theoretical total area (T) for the crude oil only chromatogram (Area $B + B'$, Fig. 3(*b*)) as follows:

$$T = [(AIS \times r) - BIS][(1 - W)/W] \quad (5)$$

where:

AIS = total area of the internal standard segment of the chromatogram of the sample plus internal standard mixture,
BIS = total area of the internal standard segment of the crude oil only chromatogram,
r = the ratio of areas outside the internal standard segment through 538°C (1000°F) for both chromatograms (see Eq 4), and
W = the mass fraction of internal standard in the mixture of crude sample plus internal standard (see Eq 3).

12.2.6 Calculate the percent residue (RES) above 538°C (1000°F) as follows:

$$RES = 100 - (B/T \times 100) \quad (6)$$

where:

B = total area through 538°C (1000°F) of the crude only chromatogram, and
T = theoretical total area of the crude oil only chromatogram (see Eq 5).

12.3 *Calculation of Boiling Point Distribution*:

12.3.1 Record the time at which the cumulative area at the beginning of the crude only chromatogram is equal to 0.5 % of the theoretical total area (T, From Eq 5). The temperature equivalent to this time is the IBP of the sample.

12.3.2 Multiply the corrected cumulative area at the end of each time interval by 100 and divide by the theoretical total area (T from Eq 5). This gives the percent of sample recovered at the end of each time interval.

12.3.3 Tabulate, in pairs, the cumulative percent recovered and the retention time at the end of each time interval.

12.3.4 Using linear interpolation where necessary, determine the time associated with each percent between 1 % and the percent eluted at the time equivalent to 538°C (1000°F).

12.3.5 For each 1 % and its associated retention time, determine the corresponding temperature from the table of boiling point-retention time calibration data (see 11.2.2).

13. Report

13.1 Report the following information:

13.1.1 The temperature to the nearest 0.5°C (1°F) at the IBP and at 1 % intervals, and

13.1.2 The total residue above 538°C to the nearest 0.1 %.

14. Precision and Bias [4]

14.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:[5]

[4] This precision was obtained from an interlaboratory cooperative study by eight laboratories on five samples. The results of this study have been filed at ASTM headquarters. Request D02-1295.

[5] API Project 44, October 31, 1972.

14.1.1 *Repeatability*—The difference between two 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, and in the normal and correct operation of the test method, exceed the values shown in Table 3 only in 1 case in 20.

14.1.2 *Reproducibility*—The difference between two single independent results obtained by different operators working in different laboratories on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the values shown in Table 3 only 1 case in 20.

NOTE 3—Samples included in the study had residues ranging from about 3 to 30 %. Samples with residues outside this range may have different precision.

TABLE 3 Repeatability and Reproducibility

% Off	Repeatability, °C	Reproducibility,° C
IBP	3.7	10.6
5	4.7	14.8
10	6.9	11.3
20	6.8	15.4
30	7.6	20.4
40	9.3	24.6
50	10.6	30.3
60	11.8	25.9
70	17.6	39.2
80	24.8	38.8
85	18.8	38.8
90	20.7	44.9
Residue	2.6 Mass %	8.1 Mass%

14.2 *Bias*—The procedure in this test method for determining the boiling range distribution of crude petroleum by gas chromatography has no bias because the boiling range distribution can only be defined in terms of a test method.

14.2.1 A rigorous, theoretical definition of the boiling range distribution of crude petroleum 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 a conventional 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.

15. Keywords

15.1 crude oil; gas chromatography; petroleum; simulated distillation

APPENDIX

(Nonmandatory Information)

X1. AGREEMENT WITH CONVENTIONAL DISTILLATION

X1.1 Test Method D2892 is the standard for conventional distillation of crude petroleum.

X1.2 Results from this test method have been compared to Test Method D2892 results by several laboratories.[6,7,8,9] Test Method D2892 has difficulty in establishing the IBP and light portion of the crude oil, and the distillation must be terminated at a maximum temperature of 400°C to prevent cracking of the sample.

X1.3 Footnote 9 is particularly significant because it shows a direct comparison of results by this test method and Test Method D2892, obtained from round-robin testing of both methods. Data from five laboratories are included.

[6] McTaggart, N. G., Glaysher, P., and Harding, A. F., *ASTM STP 577*, ASTM, 1973, p. 81.
[7] Green, L. E., "Chromatograph Gives Boiling Point," *Hydrocarbon Processing*, May 1976, p. 205.
[8] Worman, J. C., and Green, L. E., *Anal. Chem.*, Vol 37, 1965, p. 1620.
[9] Ceballo, C. D. et al. *Rev. Téc. INTEVEP*, 7(1), 1987, pp. 81–83.

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