ASTM INTERNATIONAL

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Standard Test Method for
Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Mini Method)[1]

This standard is issued under the fixed designation D7344; 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 procedure for the determination of the distillation characteristics of petroleum products and liquid fuels in the range of 20 °C to 400 °C (68 °F to 752 °F) using miniaturized automatic distillation apparatus.

1.2 This test method is applicable to such products as: light and middle distillates, automotive spark-ignition engine fuels, automotive spark-ignition engine fuels containing up to 10 % ethanol, aviation gasolines, aviation turbine fuels, all grades of No. 1 and No. 2 diesel fuels (as described in Specification D975), biodiesel (B100), biodiesel blends up to 30 % biodiesel, special petroleum spirits, pure petrochemical compounds, naphthas, white spirits, kerosenes, furnace fuel oils, and distillate marine fuels.

NOTE 1—The up to 10 % by volume ethanol limit in spark ignition engine fuels (E10) was the range used in the supporting interlaboratory studies. Spark ignition engine fuels containing > 10 % by volume ethanol and up to 20 % by volume ethanol (E20) may be analyzed, however the stated precision and bias does not apply.

1.3 This test method is designed for the analysis of distillate products; it is not applicable to products containing appreciable quantities of residual material.

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

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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
D975 Specification for Diesel Fuel Oils
D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
D5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method) (Withdrawn 2012)[3]
D5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
D5482 Test Method for Vapor Pressure of Petroleum Products (Mini Method—Atmospheric)
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

2.2 *Energy Institute Standards:*[4]
IP 69 Determination of Vapour Pressure—Reid Method
IP 394 Determination of Air Saturated Vapour Pressure

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

3. Terminology

3.1 *Definitions:*

3.1.1 *decomposition, n—of a hydrocarbon*, pyrolysis or cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule.

3.1.1.1 *decomposition point, n—in distillation*, the corrected temperature reading that coincides with the first indications of thermal decomposition of the specimen.

3.1.2 *dynamic holdup, n—in D7344 distillation*, amount of material present in the distillation column, and in the condenser during the distillation.

3.1.3 *end point (EP) or final boiling point (FBP), n—*maximum corrected temperature reading obtained during the test.

3.1.4 *initial boiling point (IBP), n—in D7344 distillation*, corrected temperature reading at the instant of the first detection of condensate in the receiver.

3.1.5 *percent evaporated, n—in distillation*, sum of the percent recovered and the percent loss.

3.1.6 *percent loss, n— in distillation*, one hundred minus the percent total recovery.

3.1.6.1 *corrected loss, n—*percent loss corrected for barometric pressure.

3.1.7 *percent recovered, n—in distillation*, the volume of condensate collected relative to the sample charge.

3.1.8 *percent recovery, n—in distillation*, maximum percent recovered relative to the sample charge.

3.1.8.1 *corrected percent recovery, n—in distillation*, the percent recovery, adjusted for the corrected percent loss.

3.1.8.2 *percent total recovery, n—in distillation*, the combined percent recovery and percent residue.

3.1.9 *percent residue, n—in distillation*, the volume of residue relative to the sample charge.

3.1.10 *sample charge, n—*the amount of sample used in a test.

3.1.11 *vapor temperature reading, n—*temperature of the saturated vapor measured in the distillation column below the vapor tube, as determined by the prescribed conditions of the test.

3.1.11.1 *corrected vapor temperature reading, n—*temperature reading, as described in 3.1.11, corrected for barometric pressure.

4. Summary of Test Method

4.1 Based on its composition, vapor pressure, expected IBP or expected FBP, or a combination thereof, the sample is placed in one of five groups. Condenser temperature and other operational variables are defined by the group in which the sample falls.

4.2 A specimen of the sample is distilled under prescribed conditions for the group in which the sample falls. The specimen volume for distillation Groups 0 to 3 is 6 mL. For Group 4, the specimen volume is 5.5 mL. The distillation is performed in an automatic, miniaturized distillation apparatus at ambient pressure under conditions that are designed to provide approximately one theoretical plate fractionation. The vapor temperature readings and volumes of condensate are monitored continuously. After the test, specimen losses and residue are recorded.

4.3 After conclusion of the test, the temperatures are automatically corrected for barometric pressure, using the pressure reading of a built-in pressure transducer. The data are examined for conformance to procedural requirements, such as distillation rates. The test has to be repeated if any specified condition has not been met.

4.4 Test results are commonly expressed as percent volume evaporated or percent volume recovered versus corresponding vapor temperature, either in a table or graphically, as a plot of the distillation curve.

4.5 This test method uses a small specimen volume and miniaturized apparatus which can be portable for field testing.

5. Significance and Use

5.1 The distillation (volatility) characteristics of hydrocarbons and other liquids have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.2 The distillation characteristics are equally important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock at high operating temperatures or high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

5.3 Volatility, as it affects the rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.

5.4 Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

5.5 This test method is suitable for setting specifications, for use as an internal quality control tool, and for use in development or research work on hydrocarbon solvents.

5.5.1 This test method gives a broad indication of general purity and can also indicate presence of excessive moisture. It will not differentiate between products of similar boiling range.

6. Apparatus

6.1 *Automatic Distillation Apparatus—*The type of apparatus suitable for this test method employs a heat source, a specimen cup, a stainless steel distillation column, a temperature measuring device, a thermoelectrically controlled condenser and receiver system, a thermoelectrically controlled sample introduction and dosing system, and a system to measure and automatically record the vapor temperature, the

associated percent recovered volume in the receiver, the condenser temperature, and the barometric pressure.[5]

6.2 A description of the apparatus is given in Annex A1.

6.3 *Sample Introduction and Dosing System*—A system capable to automatically draw sample from a sample container and fill the specimen container cup with a specimen of 6 mL ± 0.05 mL or 5.5 mL ± 0.05 mL.

6.4 *Temperature Measuring Device*—A thermocouple (NiCr-Ni or similar) in stainless steel tube of 1 mm ± 0.02 mm diameter with a response time of t(90) = 3 s ± 1 s shall be used for measuring the temperature of the vapor. The minimum resolution shall be 0.1 °C (0.2 °F), and the minimum accuracy ±0.1 °C (0.2 °F).

6.5 *Pressure Transducer*—A pressure transducer with a minimum range of 0 kPa to 120 kPa with a minimum resolution of 0.1 kPa shall be used. The minimum accuracy shall be ±0.1 kPa.

6.6 *Balance,* with a minimum range of 25 g and a minimum accuracy of ±3 mg.

6.7 *Pressure Measuring Device for Calibration,* capable of measuring local station pressure with an accuracy and a resolution of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use chemicals of at least 99 % purity for quality control checks. Quality control check materials used in this test method are toluene (**Warning—** Flammable and a health hazard) and hexadecane (see Section 10). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[6] where such specifications are available. Lower purities can be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

NOTE 2—The chemicals in this section are suggested for quality control procedures (see Section 10) and are not used for instrument calibration.

8. Sampling, Storage, and Sample Conditioning

8.1 Determine the group characteristics that correspond to the sample to be tested (see Table 1). Where the procedure is dependent upon the group, the section headings will be so marked.

8.2 *Sampling:*

TABLE 1 Group Characteristics

		Group 0	Group 1	Group 2	Group 3	Group 4
Sample characteristics		Pure	Gasoline	Gasoline	Jet Fuel	Diesel
Distillate type						
Vapor pressure at:						
37.8 °C, kPa				<65.5	<65.5	<65.5
100 °F, psi				<9.5	<9.5	<9.5
(Test Methods D323, D4953, D5190, D5191, D5482, IP 69, or IP 394)						
Distillation:						
IBP	°C	>20	≤100	>100	>100	>100
	°F	>68	≤212	>212	>212	>212
EP	°C	<400	≤250	≤250	>250	>250
	°F	<752	≤482	≤482	>482	>482

8.2.1 Only samples that are liquid at room temperature can be tested by this test method.

8.2.2 Sampling shall be done as described in Table 2 and in accordance with Practice D4057 or D4177, except do not use the "Sampling by Water Displacement" section for fuels containing oxygenates.

8.2.2.1 *Groups 1 and 2*—Collect the sample as described in 8.2.2 at a temperature below 10 °C (50 °F). If this is not possible because, for instance, the product to be sampled is at ambient temperature, the sample shall be drawn into a bottle prechilled to below 10 °C (50 °F), in such a manner that agitation is kept at a minimum. Close the bottle immediately with a tight-fitting closure. (**Warning—**Do not completely fill and tightly seal a cold bottle of sample due to the likelihood of breakage upon warming.)

8.2.2.2 *Groups 0, 3, and 4*—Collect the sample at ambient temperature. After sampling, close the sample bottle immediately with a tight-fitting closure.

8.2.2.3 If the sample received by the testing laboratory has been sampled by others and it is not known whether sampling has been performed as described in 8.2, the sample shall be assumed to have been so sampled.

8.2.2.4 Follow the manufacturer's instructions for introducing the test specimen into the measuring chamber.

8.3 *Sample Storage:*

8.3.1 If testing is not to start immediately after collection, store the samples as indicated in 8.3.2 and 8.3.3 and Table 2. All samples shall be stored away from direct sunlight or sources of direct heat.

8.3.2 *Groups 1 and 2*—Store the sample at a temperature below 10 °C (50 °F).

NOTE 3—If there are no, or inadequate, facilities for storage below or equal 10 °C (50 °F), the sample may also be stored at a temperature below 20 °C (68 °F), provided the operator ensures that the sample container is tightly closed and leak-free.

8.3.3 *Groups 0, 3, and 4*—Store the sample at ambient or lower temperature.

8.4 *Sample Conditioning Prior to Analysis:*

8.4.1 Samples shall be conditioned to the temperature shown in Table 2 before opening the sample container.

8.4.1.1 *Groups 1 and 2*—Samples shall be conditioned to a temperature of less than 10 °C (50 °F) before opening the sample container.

TABLE 2 Sampling, Storage, and Sample Conditioning

		Group 0	Group 1	Group 2	Group 3	Group 4
Temperature of sample bottle	°C		<10			
	°F		<50			
Temperature of stored bottle	°C	ambient	<10[A]	<10[A]	ambient	ambient
	°F	ambient	<50[A]	<50[A]	ambient	ambient
Temperature of sample after	°C	ambient	<10	<10	ambient	ambient
conditioning prior to analysis	°F	ambient	<50	<50	ambient	ambient
If sample is wet[B]			dry[C]	resample	resample	dry[C]	dry[C]
If sample is still wet[B]				dry[D]	dry[D]		

[A] Under certain circumstances, samples may also be stored at temperatures below 20 °C (68 °F). See also 8.3.3.
[B] If sample is known to be wet, resampling may be omitted. Dry sample in accordance with 8.5.2 and 8.5.3.
[C] Dry in accordance with 8.5.3.
[D] Dry in accordance with 8.5.2.

8.4.1.2 *Groups 0, 3, and 4*—Samples shall be conditioned to a temperature not above ambient before opening the sample container.

8.5 *Wet Samples:*

8.5.1 Samples of materials that visibly contain water are not suitable for testing. If the sample is not dry, obtain another sample that is free from suspended water.

8.5.2 *Groups 1, and 2*—If such a sample cannot be obtained, the suspended water can be removed by maintaining the sample at 0 °C to 10 °C (32 °F to 50 °F), adding approximately 10 g of anhydrous sodium sulfate per 100 mL of sample, shaking the mixture for approximately 2 min, and then allowing the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained between 1 °C and 10 °C (34 °F and 50 °F), for the analysis. Note in the report that the sample has been dried by the addition of a desiccant.

NOTE 4—Suspended water in hazy samples in Groups 1 and 2 can be removed by the addition of anhydrous sodium sulfate and separating the liquid sample from the drying agent by decanting. For Test Method D86, it has been shown that this procedure does not statistically affect the results of the test.[7]

8.5.3 *Groups 0, 3, and 4*—In cases in which a water-free sample is not practical, the suspended water can be removed by shaking the sample with anhydrous sodium sulfate or other suitable drying agent and separating it from the drying agent by decanting. Note in the report that the sample has been dried by the addition of a desiccant.

9. Preparation of Apparatus

9.1 Refer to Table 1 and prepare the instrument by choosing the appropriate group in accordance with the manufacturer's instructions.

9.2 Place a specimen cup whose mass has been determined according to the manufacturer's instructions to be known to be at least ±3 mg on the specimen cup support in the apparatus.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1455.

10. Verification of Calibration and Standardization

10.1 *Temperature Measuring Device*—Verify the calibration of the thermocouple used to monitor the vapor temperature at least every six months, and after the system has been replaced or repaired, against a thermometer which is traceable to National Institute of Standards and Technology (NIST) or national authorities in the country the equipment is used.

10.1.1 The temperature measuring device calibration can be checked by distilling pure toluene (**Warning**—Flammable and a health hazard) in accordance with this test method. The temperature measurement system shall indicate, at 50 % distilled, a temperature of 110.6 °C ± 0.8 °C (231 °F ± 1.5 °F).

10.1.2 To check the temperature measuring device at elevated temperatures, perform a distillation with hexadecane (cetane). The temperature measurement system shall indicate at 50 % distilled a temperature of 281.3 °C ± 1.5 °C (538.5 °F ± 3 °F) under Group 3 and 4 distillation conditions.

NOTE 5—The melting point of n-hexadecane is 18 °C (64.5 °F). If the sample is solid, heat it to about 25 °C (77 °F) and wait until all the material is liquid before starting the test.

10.2 *Pressure Transducer*—Check the calibration of the transducer at intervals of not more than six months, and after the instrument has been repaired. The calibration of the transducer is checked against ambient barometric pressure as measured by the pressure measuring device described in 6.7. If the displayed atmospheric pressure is not equal to the ambient barometric pressure, adjust the transducer control until the appropriate reading is observed. The ambient barometric pressure is the actual station pressure at the location of the tester at the time of measurement. (**Warning**—Many aneroid barometers, such as those used at weather stations and airports, are pre-corrected to give sea level readings. These shall not be used for calibration of the apparatus.)

10.3 *Receiver System*—The percent volume detection system of the apparatus shall have a resolution of 6 μL or better with a maximum error of 18 μL between the 0.3 mL and 6 mL points. The calibration of the assembly shall be verified in accordance with manufacturer's instructions at intervals of not more than six months and after the system has been replaced or repaired.

11. Procedure

11.1 Connect the specimen container filling system inlet tubing to the inlet of the apparatus and immerse it in the sample. For samples of Group 1, make provisions to avoid loss of high volatiles in the sample. The overall volume of the sample shall be at least 50 mL for all distillation groups (see Table 1). Follow the manufacturer's instructions for introducing the test specimen into the measuring chamber.

11.2 Regulate the temperatures of the specimen container filling system, the condenser, and the receiver system for the appropriate group of the sample as specified in Table 3. For biodiesel (B100), the condenser and receiver system is regulated to 60 °C ± 0.2 °C.

TABLE 3 Default Conditions During Test Procedure[A]

		Group 0	Group 1	Group 2	Group 3	Group 4
Temperature of filling system[A]	°C	10 ± 0.2	10 ± 0.2	10 ± 0.2	10 ± 0.2	40 ± 0.2
	°F	50 ± 0.4	50 ± 0.4	50 ± 0.4	50 ± 0.4	104 ± 0.4
Temperature of condenser[A]	°C	10 ± 0.2	10 ± 0.2	10 ± 0.2	10 ± 0.2	40 ± 0.2
	°F	50 ± 0.4	50 ± 0.4	50 ± 0.4	50 ± 0.4	104 ± 0.4
Temperature of receiver system[A]	°C	10 ± 0.2	10 ± 0.2	10 ± 0.2	10 ± 0.2	40 ± 0.2
	°F	50 ± 0.4	50 ± 0.4	50 ± 0.4	50 ± 0.4	104 ± 0.4
Time from first application of heat to initial boiling point, min		<12	<7	<8	<12	<12
Uniform rate of condensation at 5 % recovered and 95 % recovered, mL/min		≥0.15	≥0.15	≥0.15	≥0.15	≥0.15
Uniform rate of condensation from 10 % recovered to 90 % recovered, mL/min		0.3–0.9	0.3–0.9	0.3–0.9	0.3–0.9	0.3–0.9
Time from 95 % recovered to end point, min		<4	<4	<4	<4	<4
Specimen volume of sample (mL)		6 ± 0.05	6 ± 0.05	6 ± 0.05	6 ± 0.05	5.5 ± 0.05
Mean density of liquid residual, g/ccm		0.72	0.76	0.76	0.86	0.87

[A] The proper temperature will depend upon the wax content of the sample and of its distillation fractions. The test is generally performed using one single temperature. Wax formation in the condenser can be deduced from *(a)* the presence of wax particles in the distillate, *(b)* a higher distillation loss than what would be expected based on the initial boiling point of the specimen, *(c)* an erratic recovery rate and *(d)* the presence of wax particles during cleaning of the condenser with a lint-free swab. The minimum temperature that permits satisfactory operation shall be used. In some cases involving grade No. 2 fuel oil, Grade No. 2-D diesel fuel oil, gas oils, pure petrochemical compounds, and similar distillates, it may be necessary to hold the condenser, receiver and filling system temperature in the 40 °C to 60 °C (86 °F to 140 °F) range. Also, for very high volatile Group 1 samples, it may be necessary to decrease the filling and receiving temperature to 5 °C (41 °F).

11.3 *Filling*—Following the manufacturer's instructions, draw in sample, and place a specimen of 6 mL ± 0.05 mL (Groups 0 to 3) or 5.5 mL ± 0.05 mL (Group 4) into the specimen cup.

11.4 *Initial Boiling Point*—Apply heat to the specimen cup and note the start time.

11.5 Regulate the specimen container heating so that the time interval between the first application of heat and the IBP is as specified in Table 3. Record the IBP to the nearest 0.1 °C (0.2 °F).

11.6 Continue to regulate the specimen container heating so that the uniform average rate of condensation from 5 % or 10 % recovered to 95 % recovered is 0.6 mL ± 0.3 mL per min, as specified in Table 3. (**Warning**—Due to the configuration of the distillation column and the conditions of the test, the vapor and liquid around the temperature sensor are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on the measured vapor temperature. The distillation rate shall, therefore, be kept as constant as possible throughout the test.)

NOTE 6—When testing gasoline samples, it is not uncommon to see the condensate suddenly form non-miscible liquid phases and bead up on the temperature sensor and in the neck of the distillation column at a vapor temperature of around 160 °C (320 °F). This may be accompanied by a sharp (about 3 °C (6 °F)) dip in the vapor temperature and a drop in the recovery rate. The phenomenon, which may be due to the presence of trace water in the sample, may last for 10 s to 30 s before the temperature recovers and the condensate starts flowing smoothly again. This point is sometimes colloquially referred to as the Hesitation Point.

11.7 If a decomposition point is observed, discontinue the heating and proceed as directed in 11.13.

NOTE 7—Characteristic indications of thermal decomposition are evolution of fumes and erratic, typically decreasing, temperature readings that occur during the final stages of the distillation.

11.8 In the interval between the IBP and the end of the distillation, record all volumes in the receiving cylinder to the nearest 5 µL, and all temperature readings to the nearest 0.1 °C (0.2 °F).

11.8.1 Record temperature readings at least at every 0.25 % recovered.

11.9 When the percent recovered is approximately 95 %, make a final adjustment of the heat. The time from the 95 %, recovered to the EP (FBP) shall be within the limits prescribed in Table 3. If this condition is not satisfied, repeat the test with appropriate modification of the final heat adjustment.

11.10 Observe and record the EP (FBP) as required, and discontinue the heating.

11.11 Allow the distillate to drain into the receiver, after specimen heating has been discontinued. The apparatus shall continually monitor the percent recovered volume until this volume changes by no more than 10 µL in 1 min.

11.12 Record the volume in the receiver, in mL, as the percent recovered volume.

11.13 After the specimen cup has cooled place the cup on a balance and determine the mass of the cup plus residual specimen to the nearest ±3 mg. (**Warning**—Take adequate precautions when removing the sample cup to prevent accidental burns. Remove the cup in accordance with the manufacturer's instructions.)

11.13.1 If a residue larger than 300 mg is obtained and the distillation was not purposely terminated before the EP, check whether adequate heat was applied towards the end of the distillation and whether conditions during the test conform to those specified in Table 3.

11.14 Repeat any distillation that did not meet the requirements described in 11.5 and 11.6.

NOTE 8—The test method is not designed for the analysis of distillate fuels containing appreciable quantities of residual material (see 1.2).

12. Calculations

12.1 Calculate the percent recovery in accordance with the following equation:

$$R = 100\, V_R/V_S \qquad (1)$$

where:

R = percent recovery,
V_R = recovered volume, in mL (see 11.14), and
V_S = sample charge, in mL (see 11.3).

12.2 Calculate the percent residue in accordance with the following equation:

$$R_p = 100\,(M_t - M_c)/(\rho_R\, V_S) \qquad (2)$$

where:

R_p = percent residue,
M_t = total mass of specimen cup and residual specimen in the cup, as determined in 11.13, in g,
M_c = mass of the empty specimen cup, in g,
ρ_R = mean density of residual specimen (see Table 3), in g/ccm, and
V_S = sample charge, in mL (see 11.3).

12.3 The percent total recovery is the sum of the percent recovery (see 12.1) and the percent residue (see 12.2). Deduct the percent total recovery from 100 to obtain the percent loss.

12.4 Correct temperature reading to 101.3 kPa (760 mm Hg) pressure. Obtain the correction to be applied to each temperature reading by means of the Sydney Young equation as given in Eq 3, Eq 4, or Eq 5, as appropriate.

For Celsius temperatures:

$$C_c = 0.0009\,(101.3 - P_k)\,(273 + t_c) \qquad (3)$$
$$C_c = 0.00012\,(760 - P)\,(273 + t_c) \qquad (4)$$

For Fahrenheit temperatures:

$$C_f = 0.00012\,(760 - P)\,(460 + t_f) \qquad (5)$$

where:

t_c = the observed temperature reading in °C,
t_f = the observed temperature reading in °F,
C_c and C_f = corrections to be added algebraically to the observed temperature readings,
P_k = barometric pressure, prevailing at the time and location of the test, in kPa, and
P = barometric pressure, prevailing at the time and location of the test, in mm Hg.

12.4.1 After applying the corrections and rounding each result to the nearest 0.1 °C (0.2 °F), use the corrected temperature readings in all further calculations and reporting.

NOTE 9—Temperature readings are not corrected to 101.3 kPa (760 mm Hg) when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure.

12.5 Correct the actual loss to 101.3 kPa (760 mm Hg) pressure when temperature readings are corrected to 101.3 kPa pressure. The corrected loss, L_c, is calculated from Eq 6 or Eq 7, as appropriate.

$$L_c = 0.5 + (L - 0.5)/\{1 + (101.3 - P_k)/8.00\} \qquad (6)$$
$$L_c = 0.5 + (L - 0.5)/\{1 + (760 - P)/60.0\} \qquad (7)$$

where:

L = observed loss,
L_c = corrected loss,
P_k = pressure, kPa, and
P = pressure, mm Hg.

NOTE 10—Eq 6 and 7 have been derived from the data in Table 7 and Eqs 5 and 6 in Test Method D86-95 and earlier versions. It is probable that Eq 6 and 7 shown were the original empirical equations from which the table and equations in the Test Method D86-95 and earlier versions were derived.

12.5.1 Calculate the corresponding corrected percent recovery in accordance with the following equation:

$$R_c = R + (L - L_c) \qquad (8)$$

where:

L = percent loss or observed loss,
L_c = corrected loss,
R = percent recovery, and
R_c = corrected percent recovery.

12.6 To obtain the percent evaporated at a prescribed temperature reading, add the percent loss to each of the observed percent recovered at the prescribed temperature readings, and report these results as the respective percent evaporated, that is:

$$P_e = P_r + L \qquad (9)$$

where:

L = observed loss,
P_e = percent evaporated, and
P_r = percent recovered.

12.7 To report a temperature reading at a prescribed percent evaporated, obtain the desired temperature directly from the database as the temperature closest to and within 0.1 volume % of the prescribed percent evaporated.

NOTE 11—The available instruments perform most procedures described in Sections 11 and 12 automatically.

13. Report

13.1 Report the following information:

13.2 Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).

13.3 Report all volumetric readings in percentages to the nearest 0.1 %, and all temperature readings to the nearest 0.1 °C (0.2 °F).

13.4 After barometric corrections of the temperature readings have been made, the following data require no further calculation prior to reporting: IBP, EP (FBP), decomposition point, and all pairs of corresponding values involving percent recovered and temperature readings.

13.5 It is advisable to base the report on relationships between temperature readings and percent evaporated when the sample is a gasoline, or any other product classified under Group 1, or in which the percent loss is greater than 2.0. Otherwise, the report can be based on relationships between

temperature readings and percent evaporated or percent recovered. Every report must indicate clearly which basis has been used.

13.6 Report if a drying agent, as described in 8.5.2 or 8.5.3, was used.

14. Precision and Bias

14.1 *Precision*—The precision of this test method has been determined by the statistical examination of interlaboratory test results.[8]

NOTE 12—The precision has been derived according to the group number in the following fashion. Group 1 and 2 samples are labeled as NOT4, and Group 3 and 4 samples are labeled GRP4.

NOTE 13—Distillation limits are typically not specified for fuel oils of grades Nos. 4, 5, and 6.

NOTE 14—Information on the precision of % evaporated or % recovered at a prescribed temperature can be found in Annex A4.

14.1.1 *Repeatability*—The difference between successive test results obtained by 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 value only in one case in twenty:

Group 0: Refer to Annex A2 for tables of calculated repeatability.

IBP:	$r = T^3 \times 0.000006265$	valid range: 65 °C – 215 °C
T10:	$r = T^{1.6477} \times 0.0002254$	valid range: 65 °C – 240 °C
T50:	$r = T^{0.81} \times 0.004725$	valid range: 65 °C – 255 °C
T90:	$r = T^{-0.2137} \times 0.6504$	valid range: 65 °C – 255 °C
FBP:	$r = T^{0.9815} \times 0.03547$	valid range: 65 °C – 265 °C

Groups 1 and 2 (NOT4): Refer to Annex A2 for tables of calculated repeatability.

IBP:	$r = 2.46$	valid range: 20 °C – 60 °C
E10:	$r = 1.97$	valid range: 40 °C – 65 °C
E50:	$r = 0.02939 \times (E - 50)$	valid range: 75 °C – 105 °C
E90:	$r = 2.45$	valid range: 145 °C – 165 °C
FBP:	$r = 3.22$	valid range: 170 °C – 200 °C

Group 3 and 4 (GRP4): Refer to Annex A2 for tables of calculated repeatability.

IBP:	$r = 3.57$	valid range: 150 °C – 190 °C
T10:	$r = 0.04782 \times (T - 135)$	valid range: 160 °C – 220 °C
T50:	$r = 0.01926 \times (T - 137)$	valid range: 165 °C – 285 °C
T90:	$r = 0.009911 \times (T - 20)$	valid range: 180 °C – 345 °C
T95:	$r = 0.006461 \times (T + 210)$	valid range: 185 °C – 355 °C
FBP:	$r = 0.01033 \times (T - 37)$	valid range: 200 °C – 370 °C

where:
E = evaporated temperature within valid range prescribed
T = recovered temperature within valid range prescribed

NOTE 15—For naphthas, solvents, and other similar materials where percent recovered are reported and the percent loss is typically less than one percent, the percent recovered temperatures can be considered identical to the percent evaporated temperatures, and precision can be calculated as shown for Group NOT4.

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

Group 0: Refer to Annex A2 for tables of calculated reproducibility.

IBP:	$R = T^3 \times 0.0000007052$	valid range: 65 °C – 215 °C
E10:	$R = T^{1.6477} \times 0.0002509$	valid range: 65 °C – 240 °C
E50:	$R = T^{0.81} \times 0.008968$	valid range: 65 °C – 255 °C
E90:	$R = T^{-0.2137} \times 1.6210$	valid range: 65 °C – 255 °C
FBP:	$R = T^{0.9815} \times 0.06388$	valid range: 65 °C – 265 °C

Group 1 and 2 (NOT4): Refer to Annex A2 for tables of calculated reproducibility.

IBP:	$R = 5.27$	valid range: 20 °C – 60 °C
E10:	$R = 4.68$	valid range: 40 °C – 65 °C
E50:	$R = 0.04536 \times (E - 50)$	valid range: 75 °C – 105 °C
E90:	$R = 3.53$	valid range: 145 °C – 165 °C
FBP:	$R = 4.82$	valid range: 170 °C – 200 °C

Group 3 and 4 (GRP4): Refer to Annex A2 for tables of calculated reproducibility.

IBP:	$R = 5.32$	valid range: 150 °C – 190 °C
T10:	$R = 0.06407 \times (T - 135)$	valid range: 160 °C – 220 °C
T50:	$R = 0.02475 \times (T - 137)$	valid range: 165 °C – 285 °C
T90:	$R = 0.01567 \times (T - 20)$	valid range: 180 °C – 345 °C
T95:	$R = 0.01062 \times (T + 210)$	valid range: 185 °C – 355 °C
FBP:	$R = 0.01667 \times (T - 37)$	valid range: 200 °C – 370 °C

where:
E = evaporated temperature within valid range prescribed
T = recovered temperature within valid range prescribed

NOTE 16—See Note 15.

NOTE 17—Precision was not determined above E50 for one sample of aviation gasoline, because its unique boiling profile would give it undue leverage on the precision estimates. Users are cautioned that the actual precisions for aviation gasoline may differ from these estimates.

NOTE 18—The degrees of freedom associated with the reproducibility estimate from this interlaboratory study for NOT4 samples are 27 for IBP and 25 for E10. These do not meet the minimum requirements of Practice D6300. Users should be aware that the two corresponding reproducibility estimates are not as precise as the others.[10]

14.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method, bias has not been determined.

14.3 *Relative Bias*—The Degree of Agreement between results by Test Method D7344 and Test Method D86 (automated)—The 2015 interlaboratory study (ILS-1058)[8] was not used to determine the relative bias statements. The relative bias statements are from the 2005 interlaboratory study.[9] Results on the same materials produced by Test Method D7344 and Test Method D86 were assessed in accordance with procedures outlined in Practice D6708.[9] The findings were:

IBP:

NOT4

No bias-correction considered in Practice D6708 can further improve agreement between results from Test Method D7344 and Test Method D86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D6708, was observed for some samples.

Differences between results from Test Method D7344 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

The degree of agreement between results from Test Method D7344 and Test Method D86 (automated) can be further improved by applying the bias-correction outlined in Eq 10. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction.

$$Bias-corrected \: X = predicted \: Y = X - 12.4 \: °C \qquad (10)$$

where:

X = result obtained by Test Method D7344 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D86 (automated) on the same sample.

Differences between bias-corrected results from Eq 10 and Test Method D86, for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T10:

NOT4

The degree of agreement between results from Test Method D7344 and Test Method D86 (automated) can be further improved by applying the bias-correction outlined in Eq 11. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction.

$$Bias-corrected \: X = predicted \: Y = X + 3.65 \: °C \qquad (11)$$

where:

X = result obtained by Test Method D7344 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D86 (automated) on the same sample.

Differences between bias-corrected results from Eq 11 and Test Method D86, for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

The degree of agreement between results from Test Method D7344 and Test Method D86 (automated) can be further improved by applying the bias-correction outlined in Eq 12. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction.

$$Bias-corrected \: X = predicted \: Y = 0.97 \: X + 3.97 \: °C \qquad (12)$$

where:

X = result obtained by Test Method D7344 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D86 (automated) on the same sample.

Differences between bias-corrected results from Eq 12 and Test Method D86, for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T50:

NOT4

No bias-correction considered in Practice D6708 can further improve agreement between results from Test Method D7344 and Test Method D86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D6708, was observed for some samples.

Differences between results from Test Method D7344 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

The degree of agreement between results from Test Method D7344 and Test Method D86 (automated) can be further improved by applying the bias-correction outlined in Eq 13. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction.

$$Bias-corrected \: X = predicted \: Y = 0.98 \: X + 3.21 \: °C \qquad (13)$$

where:

X = result obtained by Test Method D7344 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D86 (automated) on the same sample.

Differences between bias-corrected results from Eq 13 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T90:

NOT4

No bias-correction considered in Practice D6708 can further improve agreement between results from Test Method D7344 and Test Method D86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D6708, was observed for some samples.

Differences between results from Test Method D7344 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

No bias-correction considered in Practice D6708 can further improve agreement between results from Test Method D7344 and Test Method D86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D6708, was observed for some samples.

Differences between results from Test Method D7344 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

FBP:

NOT4

No bias-correction considered in Practice D6708 can further improve agreement between results from Test Method D7344 and Test Method D86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D6708, was observed for some samples.

Differences between results from Test Method D7344 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

The degree of agreement between results from Test Method D7344 and Test Method D86 (automated) can be further improved by applying the bias-correction outlined in Eq 14. Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction.

$$\text{Bias} - \text{corrected X} = \text{predicted Y} = \text{X} - 3.81\ °\text{C} \qquad (14)$$

where:

X = result obtained by Test Method D7344 (this test method), and
bias-corrected X = predicted Y = result that would have been obtained by Test Method D86 (automated) on the same sample.

Differences between bias-corrected results from Eq 14 and Test Method D86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

NOTE 19—In some cases, stated precision for 10 % ethanol-gasoline blends at the 50 % evaporation point may not be achieved.

NOTE 20—The 2015 interlaboratory study (ILS-1058)[8] was not used to determine relative bias; users are cautioned that the actual relative bias may differ from these estimates.

14.4 The precision statements were derived according to Practice D6300 from a 2015 interlaboratory cooperative test program.[8] Eleven laboratories have participated and analyzed 29 samples comprising specification grade gasolines (some containing up to 10 % ethanol or other oxygenates), specification grade diesel (with a B7, B20, and B30 biodiesel), specification grade heating oil, aviation turbine fuels, aviation gasolines, marine fuels, mineral spirits, and toluene. The temperature range covered was 21 °C to 368 °C. The sample set also included the pure petrochemicals Hexane, Cyclohexane, Nonane, Decane, Dodecane, and Tetradecane (temperature range: 65 °C to 265 °C). Information on the type of samples and their average boiling points are in the research report.[8]

14.5 *Precision (Biodiesel B100)*[10]—The precision of this test method when applied to biodiesel (FAME) as determined by the statistical examination of the interlaboratory test results is as follows:

TABLE 4 Precision (Biodiesel B100) (ºC)

Recovery	Repeatability (r)	Reproducibility (R)	Range (°C)
IBP	0.66 (X–50)	0.66 (X–50)	155 – 245
5 %	29.4	36.9	290 – 320
10 %	19.5	26.5	305 – 335
20 %	12.8	19.4	325 – 350
30 %	5.35	7.99	330 – 360
40 % to 95 %	0.0736 (X–315)	0.1 (X–315)	335 – 370
FBP	0.395 (X–345)	0.825 (X–345)	345 – 370

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

14.5.2 *Reproducibility*—The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in normal and correct operation of this test method, exceed the values in Table 4 only in one case in twenty.

NOTE 21—The degrees of freedom associated with the reproducibility estimate from this interlaboratory study for D7344 biodiesel (B100) precision for FBP are 24. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual reproducibility may be significantly different than these estimates.

14.5.3 The precision statements for biodiesel (B100) were derived from a 2012 interlaboratory cooperative test program.[10] The precision and relative bias are applicable only to the biodiesel sample types listed in the research report. A total of nine participating laboratories using various Test Method D1160 automated, automatic, or manual apparatus; and eleven participating laboratories using D7344 apparatus; analyzed blind replicates of eleven sample sets comprised of eight specification grade biodiesel (B100) (derived from soy, canola, tallow and yellow grease), two mixed blends of biodiesel (B100) (soy and tallow), and a mustard oil. The distillation range was from 274 °C to 400 °C. Information on the type of samples and their average boiling points are in the research report.[10]

14.6 *Bias (Biodiesel B100)*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

14.7 *Relative Bias (Biodiesel B100)*—The degree of agreement between results by Test Method D7344 and Test Method D1160; results on the same materials produced by Test Method D7344 and Test Method D1160 have been assessed in accordance with procedures outlined in Practice D6708. The findings are:

14.7.1 *For IBP*—Test material property differences cannot be reliably distinguished by either Test Method D7344, or Test Method D1160, or both.

14.7.2 *For T5 to T30 %*—Test material property differences cannot be reliably distinguished by either Test Method D7344, or Test Method D1160, or both.

14.7.3 *For T40 %*—There is an insufficient degree of agreement (correlation) between Test Method D7344 and Test Method D1160.

14.7.4 *For T50 %*—The degree of agreement between results from Test Method D7344 and Test Method D1160, can be further improved by applying correction equation (Eq 15) as listed below. No sample-specific bias, as defined in Practice D6708, was observed after the bias-correction for the materials and property range studied.

$$\text{Bias-corrected X} = \text{predicted Y} = 0.67(\text{X} - 346) + 348.8\,(15)$$

where:

X = result in °C obtained by Test Method D7344 (this test method), and
bias-corrected X = predicted Y = result that would have been obtained by Test Method D1160 on the same sample.

14.7.4.1 Differences between bias-corrected results from Test Method D7344 and Test Method D1160, for the sample types and property ranges studied, are expected to exceed the

following between methods reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time. Between Methods R = $(0.5 R_X^2 + 0.5 R_Y^2)^{0.5}$ = $(0.00501 (X–315)^2 + 15.1)^{0.5}$

14.7.5 *For T60 %*—The degree of agreement between results from Test Method D7344 and Test Method D1160, can be further improved by applying correction equation (Eq 16) as listed below. No sample-specific bias, as defined in Practice D6708, was observed after the bias-correction for the materials and property range studied.

$$Bias\text{-}corrected\ X = predicted\ Y = 0.611(X - 346.9) + 349.8$$
(16)

where:

X = result in °C obtained by Test Method D7344 (this test method), and

bias-corrected X = predicted Y = result that would have been obtained by Test Method D1160 on the same sample.

14.7.5.1 Differences between bias-corrected results from Test Method D7344 and Test Method D1160, for the sample types and property ranges studied, are expected to exceed the following between methods reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time. Between Methods R = $(0.5 R_X^2 + 0.5 R_Y^2)^{0.5}$ = $(0.00501 (X–315)^2 + 10.8)^{0.5}$

14.7.6 *For T70 %*—The degree of agreement between results from Test Method D7344 and Test Method D1160, can be further improved by applying correction equation (Eq 17) as listed below. No sample-specific bias, as defined in Practice D6708, was observed after the bias-correction for the materials and property range studied.

$$bias\text{-}corrected\ X = predicted\ Y = 0.455(X - 347.9) + 350.6$$
(17)

where:

X = result in °C obtained by Test Method D7344 (this test method), and

bias-corrected X = predicted Y = result that would have been obtained by Test Method D1160 on the same sample.

14.7.6.1 Differences between bias-corrected results from Test Method D7344 and Test Method D1160, for the sample types and property ranges studied, are expected to exceed the following between methods reproducibility (R_{XY}), as defined in Practice D6708, about 5 % of the time. Between Methods R = $(0.5 R_X^2 + 0.5 R_Y^2)^{0.5}$ = $(0.00501 (X–315)^2 + 10.8)^{0.5}$

14.7.7 *For T80 %*—Test material property differences cannot be reliably distinguished by either Test Method D7344, or Test Method D1160, or both.

14.7.8 *For T90 %*—Test material property differences cannot be reliably distinguished by either Test Method D7344, or Test Method D1160, or both.

14.7.9 *For T95 %*—There is an insufficient degree of agreement (correlation) between Test Method D7344 and Test Method D1160.

14.7.10 *For FBP*—Test material property differences cannot be reliably distinguished by either Test Method D7344, or Test Method D1160, or both.

15. Keywords

15.1 atmospheric distillation; distillation; laboratory distillation; mini method; petroleum products

ANNEXES

(Mandatory Information)

A1. DESCRIPTION OF APPARATUS

A1.1 *Specimen Cup*—The specimen cup shall be made of metal (copper or aluminum have been found suitable), constructed to the dimensions and tolerances shown in Fig. A1.1.

A1.2 *Distillation, Condenser and Receiver System*—The design of the complete system is shown in Fig. A1.2.

A1.2.1 The electric heater (1) shall be of low heat retention. The power to the electric heater shall be adjustable from 0 to a minimum of 105 W.

A1.2.2 The heater is integrated in a motor driven lift (2). This lift presses the specimen cup against the bottom of the distillation column (3). The cup and the column thus form a distillation flask.

A1.2.3 The distillation column (3) shall be made of stainless steel. The thermocouple vapor temperature sensor (4) shall protrude into the distillation column from above so that the

bottom of the sensor is at the same height as the bottom of the tube connecting to the condenser.

A1.2.4 The condenser (5) shall be made of aluminum or brass. The condenser is open at the top so that the complete system is always at atmospheric pressure. The temperature of the condenser shall be controlled by a thermoelectric element and shall be adjustable from 5 °C to 60 °C (41 °F to 140 °F).

A1.2.5 The receiver system is integrated in the condenser so that condenser and receiver are always at the same temperature. The receiver consists of a meniscus detection system (6) and a variable volume receiver chamber (7) formed by a cylindrical bore hole and a piston driven by a stepper motor (8).

A1.3 *Distillation Column*—The design of the distillation column is shown in Fig. A1.3.