



Designation: D7344 – 17a

# Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Mini Method)<sup>1</sup>

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 ( $\epsilon$ ) 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, health, and environmental 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 Recom-*

*mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

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)<sup>3</sup>

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:<sup>4</sup>

IP 69 Determination of Vapour Pressure—Reid Method

IP 394 Determination of Air Saturated Vapour Pressure

<sup>1</sup> 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 D02.08 on Volatility.

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

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

\*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.<sup>5</sup>

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  $t(90) = 3 \text{ s} \pm 1 \text{ s}$  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<sup>6</sup> 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:

<sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is Grabner Instruments, A-1220 Vienna, Dr. Otto Neurathgasse 1, Austria. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>6</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

**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 <b>D323</b> , <b>D4953</b> , <b>D5190</b> , <b>D5191</b> , <b>D5482</b> , 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.