



Designation: D 86 – 90^{e2}

Standard Test Method for Distillation of Petroleum Products¹

This standard is issued under the fixed designation D 86; 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.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Paragraph 11.4.6 was corrected editorially in October 1991.

^{ε2} NOTE—Note 1 was corrected editorially in December 1992.

1. Scope

1.1 This test method covers the distillation of natural gasolines, motor gasolines, aviation gasolines, aviation turbine fuels, special boiling point spirits, naphthas, white spirit, kerosines, gas oils, distillate fuel oils, and similar petroleum products, utilizing either manual or automated equipment.

1.2 In cases of dispute, the referee test method is the manual test method prepared as directed for the indicated group.

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

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)²

D 396 Specification for Fuel Oils²

D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials³

D 975 Specification for Diesel Fuel Oils²

D 1078 Test Method for Distillation Range of Volatile Organic Liquids³

D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

D 4177 Test Method for Automatic Sampling of Petroleum and Petroleum Products⁴

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

Current edition approved Sept 28, 1990. Published November 1990. Originally published as D 86 – 21. Last previous edition D 86 – 82^{ε2}.

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 06.04.

⁴ Annual Book of ASTM Standards, Vol 05.02.

E 1 Specification for ASTM Thermometers⁵

E 77 Test Methods of Verification and Calibration of Liquid-in-Glass Thermometers⁶

E 133 Specification for Distillation Equipment⁵

E 220 Method for Calibration of Thermocouples by Comparison Techniques⁶

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *decomposition point*—the thermometer reading that coincides with the first indications of thermal decomposition of the liquid in the flask.

3.1.1.1 *Discussion*—Characteristic indications of thermal decomposition are an evolution of fumes, and erratic thermometer readings that usually decrease after any attempt is made to adjust the heat.

3.1.2 *dry point*—the thermometer reading that is observed at the instant the last drop of liquid evaporates from the lowest point in the flask. Any drops or film of liquid on the side of the flask or on the thermometer are disregarded.

3.1.2.1 *Discussion*—The end point (final boiling point), rather than the dry point, is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end point (final boiling point) whenever the sample is of such a nature that the precision of the end point (final boiling point) cannot consistently meet the requirements given in the Precision Section.

3.1.3 *end point or final boiling point*—the maximum thermometer reading obtained during the test. This usually occurs after the evaporation of all liquid from the bottom of the flask. The term *maximum temperature* is a frequently used synonym.

3.1.4 *initial boiling point*—the thermometer reading that is observed at the instant that the first drop of condensate falls from the lower end of the condenser tube.

3.1.5 *percent evaporated*—the sum of the percent recovered and the percent loss.

3.1.6 *percent loss*—one hundred minus the percent total recovery.

3.1.7 *percent recovered*—the volume in millilitres of condensate observed in the receiving graduate, in connection

⁵ Annual Book of ASTM Standards, Vol 05.03.

⁶ Annual Book of ASTM Standards, Vol 14.03.

with a simultaneous thermometer reading.

3.1.8 *percent recovery*—the maximum percent recovered, as observed in accordance with 9.10.

3.1.9 *percent residue*—the volume of residue in millilitres measured in accordance with 9.11.

3.1.10 *percent total recovery*—the combined percent recovery and residue in the flask, as determined in accordance with 9.12.

3.1.11 *thermometer reading*—the temperature of the saturated vapor measured in the neck of the flask below the vapor tube.

4. Summary of Test Method

4.1 A 100 mL sample is distilled under prescribed conditions that are appropriate to its nature. Systematic observations of thermometer readings and volumes of condensate are made, and from these data, the results of the test are calculated and reported.

5. Significance and Use

5.1 The distillation (volatility) characteristics of hydrocarbons often have an important effect on their safety and performance, especially in the case of fuels and solvents. Volatility is the major determinant of the tendency of a hydrocarbon to produce potentially explosive vapors. It is also critically important for both automotive and aviation gasolines, affecting starting, warmup, and tendency to vapor lock at high operating temperature or at 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.

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

5.3 Petroleum product specifications generally include distillation limits to assure products of suitable volatility performance.

6. Apparatus

6.1 All of the section and figure reference numbers in 6.2 through 6.8 refer to Specification E 133, the specification to which all the items listed shall conform.

6.2 *Flask*—Flask A (100 mL), as shown in Fig. 3 (of Specification E 133) for natural gasolines. Flask B (125 mL), as shown in Fig. 3 (of Specification E 133) for all others.

6.3 *Condenser and Cooling Bath*—Section 5, and Figs. 1 and 2 of Specification E 133.

6.4 *Shield*—Section 6, and Figs. 1 and 2 of Specification E 133.

6.5 *Heater*—Section 7, and Figs. 1 and 2 of Specification E 133.

6.6 *Flask Support*—Table 2 (of Specification E 133), Boards A 32-mm (1.25-in.) B 38-mm (1.5-in.) or C 50-mm (2-in.) hole.

6.7 *Graduated Cylinder*—Section 9; Graduate B, 100 mL, as shown in Fig. 4 of Specification E 133. The cylinder must have graduations at the 5 mL level and from 90 to 100 mL in 1-mL increments. For automatic apparatus, the cylinder shall conform to the physical specifications described in this section, with the exception of the graduations.

6.7.1 For automatic apparatus, the level follower/

recording mechanism of the apparatus will have a resolution of 0.1 mL with an accuracy of ± 1 mL. The calibration of the assembly should be confirmed according to manufacturer's instructions at regular intervals. The typical calibration procedure involves verifying the output with the receiver containing 5 and 100 mL of material respectively.

6.8 *Temperature Sensor*—Section 10 of Specification E 133, ASTM Thermometers 7C (7F) and 8C (8F) or IP Thermometers 5C (low distillation) and 6C (high distillation) conforming to the IP Specifications for Standard Thermometers. Under certain test conditions the bulb of the thermometer can be 28°C (50°F) above the temperature indicated, and at an indicated temperature of 371°C (700°F) the temperature of the bulb is approaching a critical range in the glass. Thermometers that have been exposed to such conditions are not to be reused without checking their ice point to verify calibration as prescribed in Specification E 1 and Test Method E 77.

6.8.1 Temperature measurement systems using thermocouples or resistance thermometers must exhibit the same temperature lag and accuracy as the equivalent mercury in glass thermometer. Confirmation of the calibration of these temperature sensors is to be made on a regular basis. This can be accomplished as described in Method E 220, potentiometrically by the use of standard precision resistance, depending on the type of probe. Another technique is to distill pure toluene in accordance with Test Method D 850 and compare the temperature indicated with that shown by the above mentioned mercury in glass thermometers when carrying out a manual test under the same conditions.

NOTE 1—Toluene is shown in reference manuals as boiling at 110.6°C under the conditions of Test Method D 1078 that uses a partial immersion thermometer. Because this test method uses total immersion thermometers the results will be lower and different with each. The approximate figures are Thermometer 7C (7F) or IP 5C at 109°C (228°F), and Thermometer 8C (8F) or IP 6C at 110°C (230°F).

7. Sampling

7.1 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.

7.2 Sampling shall be done in accordance with Practice D 4057 or Test Method D 4177 and as described in Table 2.

7.2.1 **GROUP 0**—Collect the sample in a bottle previously cooled to 0 to 4.5°C (32 to 40°F) preferably by immersing the bottle in the liquid, where possible, and discarding the first sample. Where immersion is not possible, the sample shall be drawn off into the previously cooled bottle in such a manner that agitation is kept at a minimum. Close the bottle immediately with a tight-fitting stopper and place the sample in an ice bath or refrigerator to maintain the sample at that temperature.

7.2.2 **GROUPS 1 and 2**—Collect and maintain the sample as described in 7.2.1 at a temperature of 0 to 10°C (32 to 50°F).

7.2.3 **GROUPS 3 and 4**—Maintain the sample at ambient temperature. If sample is not fluid at ambient temperature, it is to be maintained at a temperature of 11°C (20°F) above its pour point.

7.3 Samples of materials that visibly contain water are not suitable for testing.

TABLE 1 Group Characteristics

	Group 0	Group 1	Group 2	Group 3	Group 4
Sample Characteristics:					
Distillate Type:	Natural Gasoline				
Vapor pressure at 37.8°C, kPa		≥ 65.5	< 65.5	< 65.5	< 65.5
100°F, psi		≥ 9.5	< 9.5	< 9.5	< 9.5
(Test Method D 323 IP 69 or IP 171)					
Distillation, IPB °C				≤ 100	> 100
°F				≤ 212	> 212
EP °C		≤ 250	≤ 250	> 250	> 250
°F		≤ 482	≤ 482	> 482	> 482

TABLE 2 Sampling

	Group 0	Group 1	Group 2	Group 3	Group 4
Temperature of Sample Bottle: °C	0 to 4.5	0 to 10			
°F	32 to 40	32 to 50			
Temperature of Stored Sample: °C	0 to 4.5	0 to 10	0 to 10	Ambient	Ambient
°F	32 to 40	32 to 50	32 to 50	11°C above pour point	11°C above pour point
				Ambient	Ambient
				20°F above pour point	20°F above pour point
If sample is wet:	Resample	Resample	Resample	Dry in accordance with 7.3.2	Dry in accordance with 7.3.2

TABLE 3 Preparation of Apparatus

	Group 0	Group 1	Group 2	Group 3	Group 4
Flask, mL	100	125	125	125	125
ASTM Distillation Thermometer	7C (7F)	7C (7F)	7C (7F)	7C (7F)	8C (8F)
IP Distillation Thermometer	5C	5C	5C	5C	6C
Flask Support	A	B	B	C	C
Diameter of hole, mm (in.)	32 (1.25)	38 (1.5)	38 (1.5)	50 (2.0)	50 (2.0)
Temperature at start of Test:					
Flask and thermometer, °C	0 to 4.5	13 to 18	13 to 18	13 to 18	Not above
°F	32 to 40	55 to 65	55 to 65	55 to 65	ambient
Flask support and shield	Not above ambient	Not above ambient	Not above ambient	Not above ambient	...
Graduate and 100 mL charge, °C	0 to 4.5	13 to 18	13 to 18	13 to 18	13 to ambient
°F	32 to 40	55 to 65	55 to 65	55 to 65	55 to ambient

7.3.1 GROUPS 0, 1, and 2—If the sample is not dry, obtain another sample that is free from suspended water for the test.

7.3.2 GROUPS 3 and 4—In cases where 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.

8. Preparation of Apparatus

8.1 Refer to Table 3 and prepare the apparatus as directed for the indicated group. Bring the temperature of the graduate, the flask, the temperature sensor, and the cooling bath to the indicated temperature.

8.2 Make any necessary provisions so that the temperature of the cooling bath and the graduate will be maintained at their respective temperatures. The cooling bath must have a liquid level above the highest point of the condenser. If necessary, make suitable provision for circulation, stirring, or air blowing to provide a uniform temperature throughout the bath. The graduate must be in a bath such that either the liquid level is at least as high as the 100 mL mark, or the entire graduate is surrounded by an air circulation chamber.

8.2.1 GROUPS 0, 1, 2, and 3—Suitable media for low temperature baths include chopped ice and water, refrigerated brine, and refrigerated ethylene glycol.

8.2.2 GROUP 4—Suitable media for ambient and higher

bath temperatures can include cold water, hot water, or heated ethylene glycol.

8.3 Remove any residual liquid in condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or copper wire.

8.4 GROUPS 0, 1, 2, and 3—Fit a thermometer 7C (7F), provided with a snug-fitting, well-rolled cork or silicone-rubber stopper, tightly into the neck of the sample container and bring the temperature of the sample to the temperature indicated in Table 3.

8.5 Measure 100 mL of sample in the graduate and transfer as completely as practical the contents of the graduate to the distillation flask, taking care that none of the liquid flows into the vapor tube.

8.6 Fit the temperature sensor, provided with a snug-fitting, well-rolled cork or silicone-rubber stopper, tightly into the neck of the flask. In the case of a thermometer, the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapor tube (see Fig. 1). In the case of thermocouple/resistance thermometer, follow the manufacturer's instructions as to placement.

8.7 Fit the flask vapor tube, provided with a snug-fitting, well-rolled cork or silicone rubber stopper, tightly into the condenser tube. Adjust the flask in a vertical position and so that the vapor tube extends into the condenser tube for a distance of 25 to 50 mm (1 to 2 in). Raise and adjust the

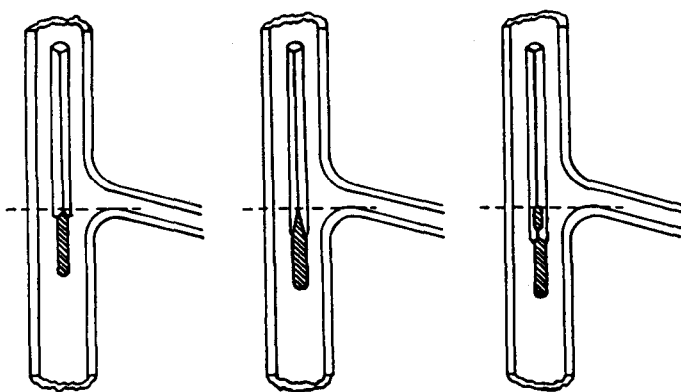


FIG. 1 Position of Thermometer in Distillation Flask

flask support board to fit snugly against the bottom of the flask.

8.8 Place the graduate that was used to measure the charge, without drying, into its bath under the lower end of the condenser tube so that the end of the condenser tube is centered in the graduate and extends therein for a distance of at least 25 mm (1 in), but not below the 100-mL mark. Cover the graduate closely with a piece of blotting paper, or similar material, that has been cut to fit the condenser tube snugly.

8.9 Record the room temperature and prevailing barometric pressure. Proceed at once with the distillation, as given in the Procedure Section.

9. Procedure

9.1 Apply heat to the distillation flask and contents. The heating at this stage must be so regulated that the time interval between the first application of heat and the initial boiling point is as indicated in Table 4.

9.2 Observe and record the initial boiling point. If a receiver deflector is not being used, immediately move the graduate so that the tip of the condenser touches its inner wall.

9.3 Regulate the heating so that the time from initial

boiling point to 5 or 10 % recovered is as indicated in Table 4.

9.4 Continue to regulate the heating so that the uniform average rate of condensation from 5 or 10 % recovered to 5 mL residue in the flask is 4 to 5 mL per min.

9.5 Repeat any distillation that did not meet the foregoing conditions.

9.6 If a decomposition point is observed, discontinue the heating and proceed as directed in 9.10.

9.7 In the interval between the initial boiling point and the end of the distillation, observe and record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the sample under test. These observed data can include thermometer readings at prescribed percentages recovered, or percentages recovered at prescribed thermometer readings, or both. Record all volumes in the graduate to the nearest 0.5 or 0.1 mL, and all thermometer readings to the nearest 0.5°C (1.0°F) or 0.1°C (0.1°F) as appropriate to the apparatus being used.

9.7.1 **GROUP 0**—In cases in which no specific data requirements have been indicated, record the initial boiling point, the end point (final boiling point) and thermometer readings at each 10 % multiple of volume recovered from 10 to 90, inclusive.

9.7.2 **GROUP 1, 2, 3, and 4**—In cases in which no specific data requirements have been indicated, record the initial boiling point, the end point (final boiling point) or dry point, or both, and thermometer readings at 5, 15, 85 and 95 % recovered, and at each 10 % multiple of volume recovered from 10 to 90, inclusive.

9.8 When the residual liquid in the flask is approximately 5 mL, make a final adjustment of the heat so that the time from the 5 mL of liquid residue in the flask to the end point (final boiling point) shall be within the limits prescribed in Table 4. If this condition is not satisfied, repeat the test, with appropriate modification of the final heat adjustment.

9.9 Observe and record the end point (final boiling point) or dry point, or both, as required, and discontinue the heating.

9.10 While the condenser tube continues to drain into the

TABLE 4 Conditions During Test Procedure

	Group 0	Group 1	Group 2	Group 3	Group 4
Temperature of cooling bath ^A , °C	0 to 1	0 to 1	0 to 4	0 to 4	0 to 60
°F	32 to 34	32 to 34	32 to 40	32 to 40	32 to 140
Temperature of bath around graduate, °C	0 to 4	13 to 18	13 to 18	13 to 18	± 3
°F	32 to 40	55 to 65	55 to 65	55 to 65	± 5 of charge temperature
Time from first application of heat to initial boiling point, minutes	2 to 5	5 to 10	5 to 10	5 to 10	5 to 15
Time from initial boiling point to 5 % recovered, seconds	...	60 to 75	60 to 75	...	...
to 10 % recovered, minutes	3 to 4				
Uniform average rate of condensation from 5 % recovered to 5 mL residue in flask, mL/min	4 to 5	4 to 5	4 to 5	4 to 5	4 to 5
Time recorded from 5 mL residue to end point, min	3 to 5	3 to 5	3 to 5	5 max	5 max

^A The proper condenser bath temperature will depend upon the wax content of the sample and of its distillation fractions. The minimum temperature that permits satisfactory operation shall be used. In general, a bath temperature in the 0 to 4°C (32 to 40°F) range is suitable for kerosine and products meeting the specifications for Grade No. 1 fuel oil as prescribed in Specification D 396, and those meeting the specifications for Grade No. 1-D diesel fuel oil as prescribed in Specification D 975. In some cases involving Grade No. 2 fuel oil (see Specification D 396), Grade No. 2-D diesel fuel oil (see Specification D 975), gas oils and similar distillates, it may be necessary to hold the condenser bath temperature at some point in the 38 to 60°C (100 to 140°F) range, in order to avoid the condensation of solid wax materials in the condenser tube.