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ASTM STANDARD D86: "STANDARD TEST METHOD FOR DISTILLATION OF PETROLEUM PRODUCTS."

by

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Designation: D 86 – 95

An American National Standard

Standard Test Method for Distillation of Petroleum Products[1]

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.

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 only.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)[2]
D 396 Specification for Fuel Oils[2]
D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials[3]
D 975 Specification for Diesel Fuel Oils[2]
D 1078 Test Method for Distillation Range of Volatile Organic Liquids[3]
D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)[4]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[4]
D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline Oxygenate Blends (Dry Method)[5]
D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)[5]
D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)[5]
D 5482 Test Method for Vapor Pressure of Petroleum Products (Mini Method-Atmospheric)[5]
E 1 Specification for ASTM Thermometers[6]
E 77 Test Method for Inspection and Verification of Thermometers[6]
E 133 Specification for Distillation Equipment[7]
E 220 Method for Calibration of Thermocouples by Comparison Techniques[6]
IP 69 Determination of Vapour Pressure–Reid Method[8]
IP 171 Vapour Pressure Micro Method[9]

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.

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.

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.

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[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.

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[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.

[5] *Annual Book of ASTM Standards*, Vol 05.03.

[6] *Annual Book of ASTM Standards*, Vol 14.03.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

[8] Available from The Institute of Petroleum, 61 New Cavendish St., London, W1M 8AR, England.

[9] Withdrawn 1992, contact The Institute of Petroleum for availability.

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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 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 Unless noted otherwise, 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. 1a of this test method or 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.

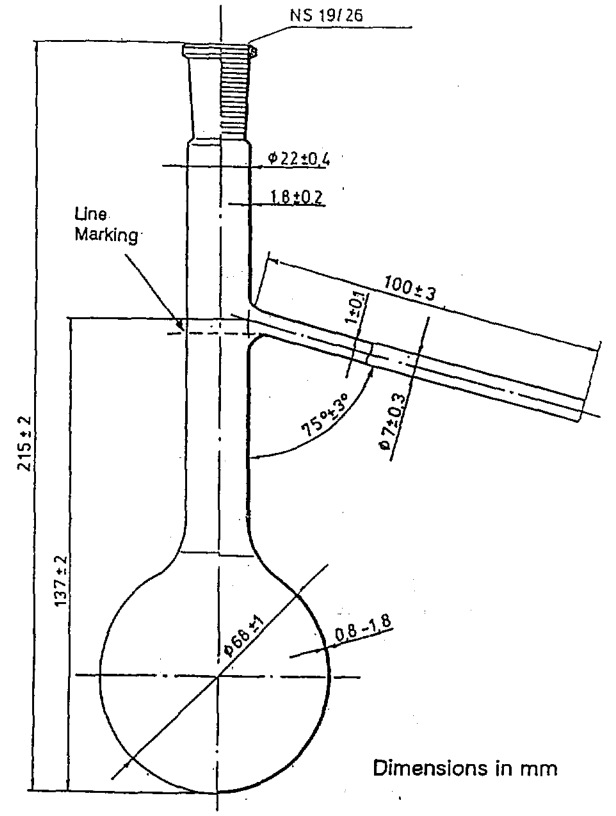

FIG. 1a Distillation Flask with Ground Glass Joint

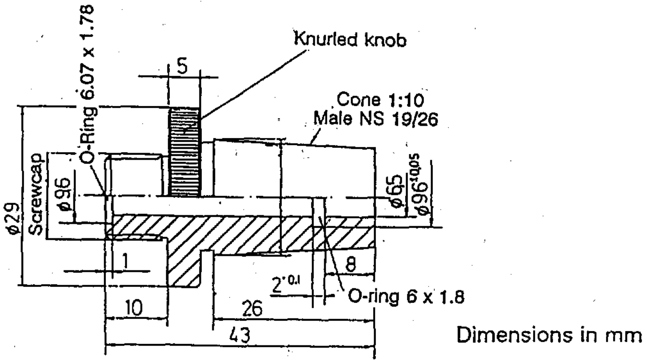

FIG. 1b PTFE Centering Device for Ground Glass Joint

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)

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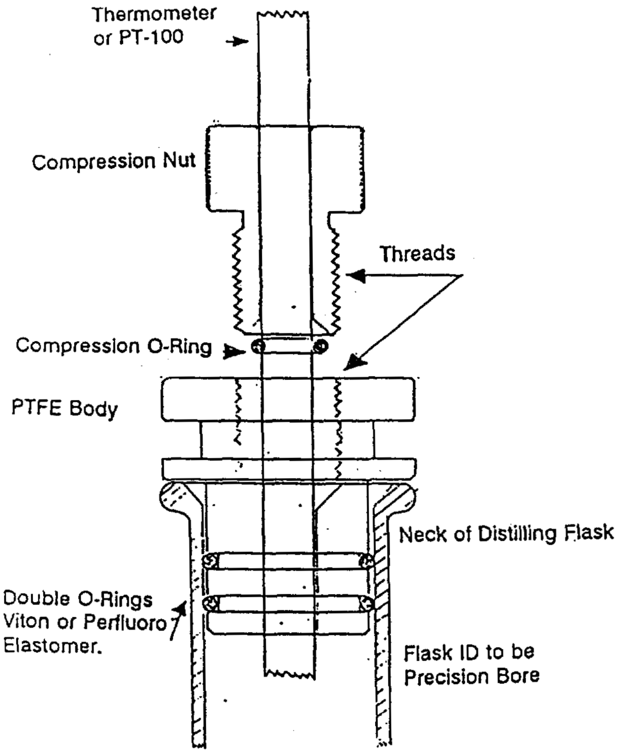

FIG. 2 Centering Device for Straight-Bore Neck

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—When running the test by the manual method, products with a low initial boiling point may have one or more readings obscured by the centering device.

NOTE 2—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).

6.8.2 The temperature sensor shall be mounted through a snug-fitting device designed to mechanically center the sensor in the neck of the flask. The use of a cork or silicone stopper with a hole drilled through the center is not acceptable for this purpose. Examples of acceptable centering devices are shown in Figs. 1 and 2.

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.

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

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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 Methods D 323, D 4953, D 5190, D 5191, D 5482, 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 11°C above pour point	Ambient
	°F	32 to 40	32 to 50	32 to 50	Ambient 20°F above pour point	Ambient
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 ambient
°F	32 to 40	55 to 65	55 to 65	55 to 65	
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

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 through a snug-fitting device designed to mechanically center the sensor in 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. 3). 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 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.

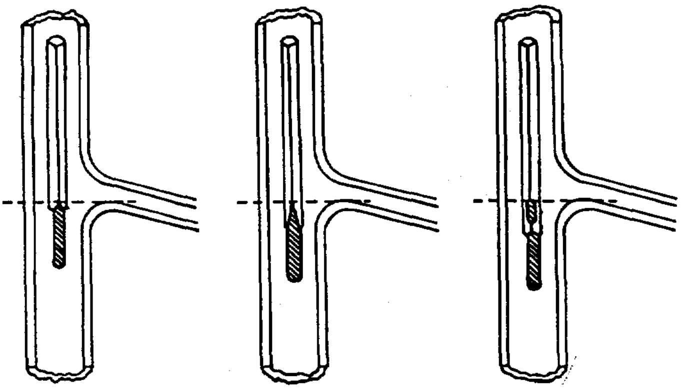

FIG. 3 Position of Thermometer in Distillation Flask

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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 graduate, observe the volume of condensate at 2 min intervals until two successive observations agree. Measure this volume accurately, and record it, to the nearest 0.5 or 0.1 mL as appropriate to the apparatus being used, as percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovery from 100, report this difference as percent residue and loss, and omit the procedure given in 9.11.

9.11 After the flask has cooled, pour its contents into a 5 mL graduated cylinder, and with the flask suspended over the 5 mL graduate, allow the flask to drain until no appreciable increase in the volume of liquid in the 5 mL graduate is observed.

9.11.1 **GROUP 0**—Cool the graduate to 0 to 4.5°C (32 to 40°F). Record the volume in the graduate, to the nearest 0.1 mL, as percent residue.

9.11.1 **GROUPS 1, 2, 3, and 4**—Record the volume in the graduate, to the nearest 0.1 mL, as percent residue.

9.12 The sum of the percent recovery (see 9.10) and the percent residue (see 9.11) is the percent total recovery. Deduct the percent total recovery from 100 to obtain the percent loss.

10. Calculations and Report

10.1 For each test, calculate and report whatever data are required by the specification involved, or as previously established for the sample under test (see 9.7). Report all percentages to the nearest 0.5 or 0.1, 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. Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).

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 waxy materials in the condenser tube.

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10.2 **GROUP 4**—When ASTM Thermometer 8C (8F) or IP Thermometer 6C is used in testing aviation turbine fuels and similar products, pertinent thermometer readings can be obscured by the cork. To provide the desired data, a second distillation according to Group 3 may have to be performed. In such cases, reading from ASTM thermometer 7C (7F) or IP Thermometer 5C can be reported in place of the obscured ASTM Thermometer 8C (8F) or IP Thermometer 6C readings, and the test report shall so indicate. If, by agreement, the obscured readings are waived, the test report shall so indicate.

10.3 Thermometer readings shall be corrected to 101.3 kPa (760 mm Hg) pressure except when product definitions, specifications, or agreements between the purchaser and the seller indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure. This report shall include the observed pressure and shall state whether corrections have or have not been applied. When the report is based on thermometer readings corrected to 101.3 kPa (760 mm Hg), obtain the correction to be applied to each thermometer reading by means of the Sydney Young equation as given in Eq. 1, or by the use of Table 5. For Celsius temperatures:

$$C_c = 0.0009\,(101.3 - P_k)(273 + t_c) \tag{1}$$

$$C_c = 0.00012\,(760 - P)(273 + t_c) \tag{2}$$

For Fahrenheit temperatures:

$$C_f = 0.00012\,(760 - P)(460 + t_f) \tag{3}$$

where:

C_c and C_f = corrections to be added algebraically to the observed thermometer readings t_c or t_f, respectively,
P_k = barometric pressure, kPa, prevailing at the time and location of the test, and
P = barometric pressure, mm Hg, prevailing at the time and location of the test.

After applying the corrections and rounding each result to the nearest 0.5°C (1.0°F) or 0.1°C (0.1°F) as appropriate to the apparatus being used, use the corrected thermometer readings in all further calculations and reporting.

TABLE 5 Approximate Thermometer Reading Corrections

Temperature Range		Correction[A] per 1.3 kPa (10 mm) Difference in Pressure	
°C	°F	°C	°F
10 to 30	50 to 86	0.35	0.63
30 to 50	86 to 122	0.38	0.68
50 to 70	122 to 158	0.40	0.72
70 to 90	158 to 194	0.42	0.76
90 to 110	194 to 230	0.45	0.81
110 to 130	230 to 266	0.47	0.85
130 to 150	266 to 302	0.50	0.90
150 to 170	302 to 338	0.52	0.94
170 to 190	338 to 374	0.54	0.99
190 to 210	374 to 410	0.57	1.03
210 to 230	410 to 446	0.59	1.06
230 to 250	446 to 482	0.62	1.12
250 to 270	482 to 518	0.64	1.15
270 to 290	518 to 554	0.66	1.19
290 to 310	554 to 590	0.69	1.24
310 to 330	590 to 626	0.71	1.28
330 to 350	626 to 662	0.74	1.33
350 to 370	662 to 698	0.76	1.37
370 to 390	698 to 734	0.78	1.40
390 to 410	734 to 770	0.81	1.46

[A] To be added when barometric pressure is below 101.3 kPa (760 mm Hg); to be subtracted when barometric pressure is above 101.3 kPa (760 mm Hg).

10.4 After barometric corrections of the thermometer reading readings have been made, if required (see 10.3), the following data require no further calculation prior to reporting: initial boiling point, dry point, end point (final boiling point), decomposition point, and all pairs of corresponding values involving percentages recovered and thermometer readings.

10.5 When thermometer readings are corrected to 101.3 kPa (760 mm Hg) pressure, the actual loss shall be corrected to 101.3 kPa (760 mm Hg) pressure, according to the following equation:

$$L_c = AL + B \tag{4}$$

where:

L = percent loss as calculated from test data,
L_c = corrected loss, and
A and B = numerical constants.

10.5.1 The values of A and B that depend upon the prevailing barometric pressure are listed in Table 6. The following equation can be substituted:

$$L_c = \{(L - 0.499287)/(13.65651 - 0.12492914\,P_k)\} + 0.4997299 \tag{5}$$

$$L_c = \{(L - 0.499287)/(13.65651 - 0.01665174\,P)\} + 0.4997299 \tag{6}$$

where:

L = percent loss as calculated from test data,
L_c = corrected loss,
P_k = pressure, kPa, and
P = pressure, mm Hg.

10.5.2 The corresponding corrected percent recovery is calculated according to the following equation:

$$R_c = R + (L - L_c) \tag{7}$$

where:

L = observed loss,
L_c = corrected loss,
R = observed recovery, and

TABLE 6 Values of Constants A and B Used in Obtaining Corrected Distillation Loss

Observed Barometric Pressure			
kPa	mm Hg	A	B
74.6	560	0.231	0.384
76.0	570	0.240	0.380
77.3	580	0.250	0.375
78.6	590	0.261	0.369
80.0	600	0.273	0.363
81.3	610	0.286	0.357
82.6	620	0.300	0.350
84.0	630	0.316	0.342
85.3	640	0.333	0.333
86.6	650	0.353	0.323
88.0	660	0.375	0.312
89.3	670	0.400	0.300
90.6	680	0.428	0.286
92.0	690	0.461	0.269
93.3	700	0.500	0.250
94.6	710	0.545	0.227
96.0	720	0.600	0.200
97.3	730	0.667	0.166
98.6	740	0.750	0.125
100.0	750	0.857	0.071
101.3	760	1.000	0.000

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R_c = corrected recovery.

10.5.3 When the thermometer readings have not been corrected to 101.3 kPa (760 mm Hg) pressure, the percent residue and percent loss are to be reported as observed in accordance with 9.11 and 9.12 respectively.

10.5.4 When reporting data, state whether the corrections have or have not been applied.

10.5.5 The corrected loss shall not be used in the calculation of percentages evaporated.

10.6 It is advisable to base the report on relationships between thermometer readings and percentages evaporated in any case in which the sample is a gasoline, or any other product classed under **GROUP 1**, or in which the percent loss is greater than 2.0. Otherwise, the report can be based on relationships between thermometer readings and percentages evaporated or recovered. Every report must indicate clearly which basis has been used.

10.7 To report percentages evaporated at prescribed thermometer readings, add the percent observed loss to each of the observed percentages recovered at the prescribed thermometer readings, and report these results as the respective percentages evaporated, that is:

$$P_e = P_r + L \tag{8}$$

where:

L = observed loss,

P_e = percentage evaporated, and

P_r = percentage recovered.

10.8 To report thermometer readings at prescribed percentages evaporated, use either of the two following procedures, and indicate on the report whether the graphical procedure or the arithmetical procedure has been used.

10.8.1 *Arithmetical Procedure*—Deduct the observed distillation loss from each prescribed percentage evaporated in order to obtain the corresponding percentage recovered. Calculate each required thermometer reading as follows:

$$T = T_L + \frac{(T_H - T_L)(R - R_L)}{R_H - R_L} \tag{9}$$

where:

R = percent recovered corresponding to the prescribed percent evaporated,

R_H = percent recovered adjacent to, and higher than R,

R_L = percent recovered adjacent to, and lower than R,

T = thermometer reading at the prescribed percent evaporated.

T_H = thermometer reading recorded at R_H and

T_L = thermometer reading recorded at R_L.

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are nonlinear. Intervals between successive data points can, at any stage of the test, be no wider than the intervals indicated in 9.7. In no case shall a calculation be made that involves extrapolation.

10.8.2 *Graphical Procedure*—Using graph paper with uniform subdivisions, plot each thermometer reading corrected for barometric pressure, if required (see 10.3), against its corresponding percent recovered. Plot the initial boiling point at 0 % recovered. Draw a smooth curve connecting the points. For each prescribed percent evaporated, deduct the distillation loss, in order to obtain the corresponding percent recovered, and take from the graph the thermometer reading which this percent recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

TABLE 7 Repeatability and Reproducibility for Group 1 (Manual)

Evaporated Point	Repeatability[A] °C	Repeatability[A] °F	Reproducibility[A] °C	Reproducibility[A] °F
IBP	3.3	6	5.6	10
5 %	$r_0 + 0.66$	$r_0 + 1.2$	$R_0 + 1.11$	$R_0 + 2.0$
10 to 80 %	r_0	r_0	R_0	R_0
90 %	r_0	r_0	$R_0 - 1.22$	$R_0 - 2.2$
95 %	r_0	r_0	$R_0 - 0.94$	$R_0 - 1.7$
FBP	3.9	7	7.2	13

[A] Read r_0 and R_0 from the graph in either Fig. 4 (°C) or Fig. 5 (°F).

NOTE 3—See Appendix X1 for numerical examples illustrating these arithmetical procedures.

11. Precision and Bias

11.1 The precision of this test method as determined by the statistical examination of interlaboratory test results is described below. Table A1.1 lists which tables and figures are to be used for the different fuel groups, distillation methods, and temperature scales.

11.2 *Repeatability:*

11.2.1 **GROUP 0**—With proper care and strict attention to details, duplicate results obtained for endpoint should not differ from each other by more than 3.5°C (6°F). Differences in duplicate temperature readings for each prescribed percentage point should not exceed the amounts equivalent to 2 mL of distillate at each point in question.

11.2.2 **GROUP 1**—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated in Table 7 (Manual) or Table 8 (Automatic) in one case in twenty.

11.2.3 **GROUPS 2, 3, and 4**—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would in the normal and correct operation of this test method, exceed the values indicated in Fig. 8 (Manual, °C) or Fig. 9 (Manual, °F) or Table 9 (Automatic) in 1 case in 20.[10,11]

11.3 *Reproducibility:*

11.3.1 **GROUP 1**—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would in the normal and correct operation of this test method, exceed the values indicated in Table 7 (Manual) or Table 8 (Automatic) in one case in twenty.

11.3.2 **GROUPS 2, 3, and 4**—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would in the normal and correct operation of this test method, exceed the values indicated in Fig. 6 (Manual, °C) or Fig. 8 (Manual, °F) or Table 9 (Automatic) in only one case in twenty.[10,11]

11.4 To facilitate the use of the tables and figures, the rate

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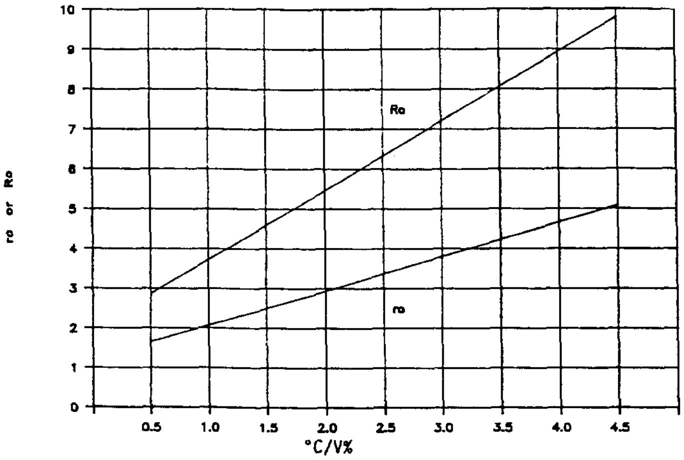
[10] Test Method D 86 Manual Method North American and IP Labs.

[11] Test Method D 86 Automatic Method North American and IP Labs.

Attachment 9

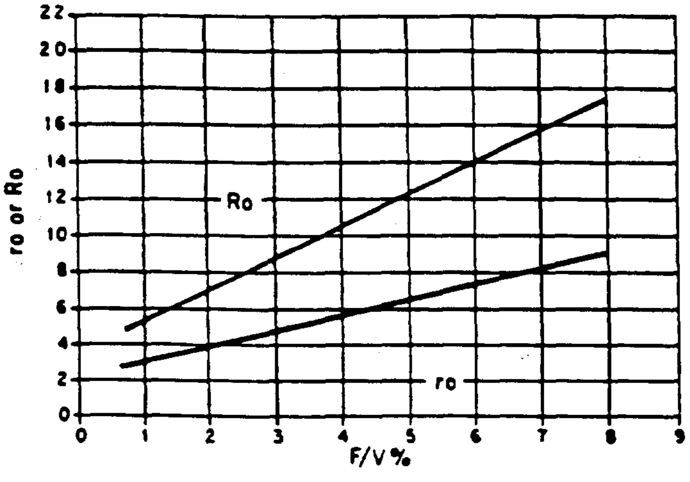


NOTE—

$$r_0 = 0.864\ (°\text{C/V}\ \%) + 1.214 \quad (14)$$

$$R_0 = 1.736\ (°\text{C/V}\ \%) + 1.994 \quad (15)$$

FIG. 4 GROUP 1—Repeatability, r_0 and Reproducibility, R_0[10]



NOTE—

$$r_0 = 0.864\ (°\text{F/V}\ \%) + 2.186 \quad (16)$$

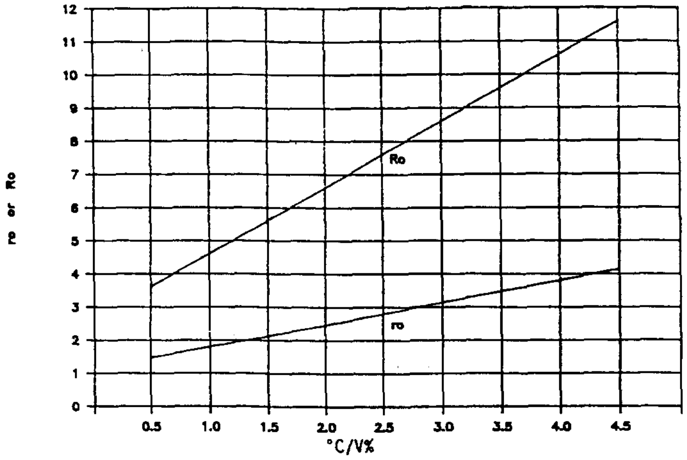
$$R_0 = 1.736\ (°\text{F/V}\ \%) + 3.589 \quad (17)$$

FIG. 5 GROUP 1—Repeatability, r_0 and Reproducibility, R_0[10]

TABLE 8 Repeatability and Reproducibility for Group 1 (Automatic)

Evaporated Point	Repeatability[A] °C	Repeatability[A] °F	Reproducibility[A] °C	Reproducibility[A] °F
IBP	3.9	7	7.2	13
5 %	r_0 + 1.0	r_0 + 1.8	R_0 + 1.78	R_0 + 3.2
10 %	r_0 + 0.56	r_0 + 1.0	R_0 + 0.72	R_0 + 1.3
20 %	r_0	r_0	R_0 + 0.72	R_0 + 1.3
30 to 70 %	r_0	r_0	R_0	R_0
80 %	r_0	r_0	R_0 − 0.94	R_0 − 1.7
90 %	r_0	r_0	R_0 − 1.9	R_0 − 3.5
95 %	r_0 + 1.4	r_0 + 2.5	R_0	R_0
FBP	4.4	8	8.9	16

[A] Read r_0 and R_0 from the graph in either Fig. 6 (°C) or Fig. 7 (°F).

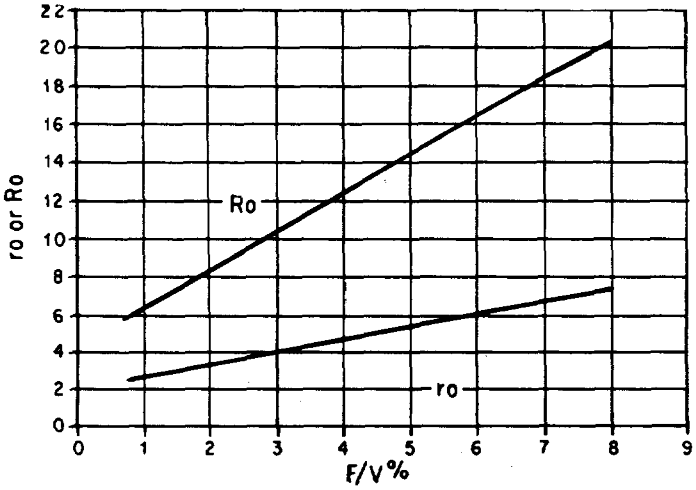


NOTE—

$$r_0 = 0.673\ (°\text{C/V}\ \%) + 1.131 \quad (18)$$

$$R_0 = 1.998\ (°\text{C/V}\ \%) + 2.617 \quad (19)$$

FIG. 6 GROUP 1—Repeatability, r_0 and Reproducibility, R_0[11]



NOTE—

$$r_0 = 0.673\ (°\text{F/V}\ \%) + 2.036 \quad (20)$$

$$R_0 = 1.998\ (°\text{F/V}\ \%) + 4.711 \quad (21)$$

FIG. 7 GROUP 1—Repeatability, r_0 and Reproducibility, R_0[11]

TABLE 9 Repeatability and Reproducibility for Groups 2, 3, and 4 (Automatic)

% Collected	Repeatability[A] °C	Repeatability[A] °F	Reproducibility[A] °C	Reproducibility[A] °F
IBP	3.5	6.3	8.5	15.3
2 %	3.5	6.3	2.6 + 1.92 *S*	4.7 + 1.92 *S*
5 %	1.1 + 1.08 *S*	2.0 + 1.08 *S*	2.0 + 2.53 *S*	3.6 + 2.53 *S*
10 %	1.2 + 1.42 *S*	2.2 + 1.42 *S*	3.0 + 2.64 *S*	5.4 + 2.64 *S*
20 to 70 %	1.2 + 1.42 *S*	2.2 + 1.42 *S*	2.9 + 3.97 *S*	5.2 + 3.97 *S*
80 %	1.2 + 1.42 *S*	2.2 + 1.42 *S*	3.0 + 2.64 *S*	5.4 + 2.64 *S*
90 to 95 %	1.1 + 1.08 *S*	2.0 + 1.08 *S*	2.0 + 2.53 *S*	3.6 + 2.53 *S*
FBP	3.5	6.3	10.5	18.9

[A] *S* is the average slope calculated in accordance with 11.4.

of change in thermometer readings in degrees Celsius (Fahrenheit) per the percentage recovered, at any point between the 10 and 90 % point, is assumed to be the same as the average rate between two data points that are equidistant above and below the point in question. The span from the point in question to either of the other data points does not represent more than 10 % recovered in any case, nor more than 5 % if the point in question is the 5 % point. Precision values for typical values of slope for **GROUPS 2, 3, and 4** (Automatic) are given in Table 10.

11.4.1 The equations that follow are used as guidelines in calculating *C/V %* (*F/V %*). In the event the distillation end point occurs prior to the 95 % point, then appropriate modifications to the use of these equations must be made.

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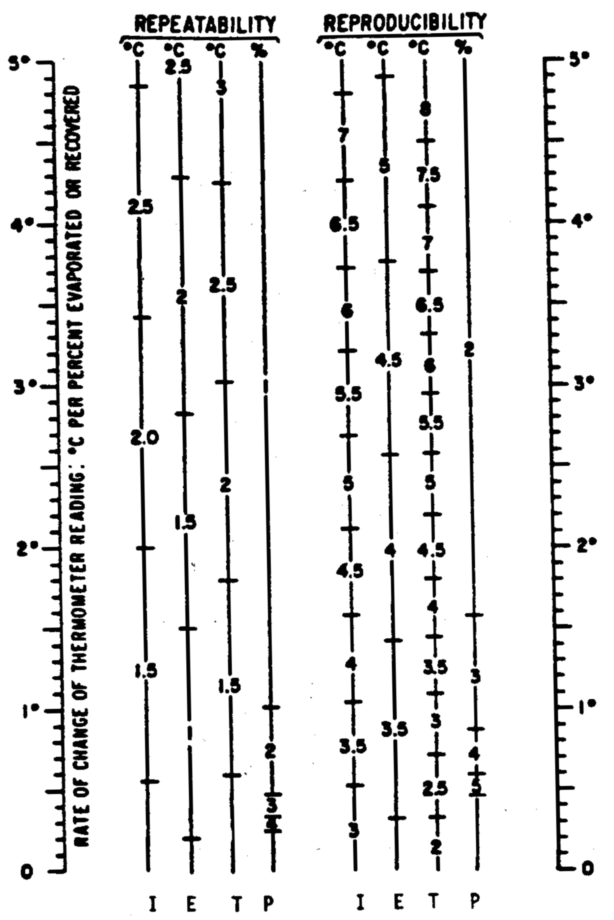


NOTE—*I* = initial boiling point, °C,
E = end point (final boiling point) or dry point, °C,
T = thermometer reading at prescribed percent evaporated or recovered, °C, and
P = percent evaporated or recovered at prescribed thermometer reading, °C.

FIG. 8 Groups 2, 3, 4—Manual Method-Celsius Precision of Distillation Test Method D 86 - IP 123

11.4.2 For Group 1 in the manual method and for all groups in the automatic method, the initial boiling point and end point do not require *C/V %* (*F/V %*).

11.4.3 For Groups 2, 3, and 4 in the manual method, the rate of change in thermometer readings in degrees Celsius per percentage recovered should be calculated from the nearest reading (5 % in the case of the IBP, and either 90 or 95 % in the case of FBP) and the corresponding IBP or FBP.

11.4.4 5 % recovered is calculated as follows:

$$C/V\%,\ F/V\% = 0.1(T_{10} - T_{IBP}) \qquad (10)$$

11.4.5 10 to 80 % recovered is calculated as follows:

$$C/V\%,\ F/V\% = 0.05(T_{(V+10)} - T_{(V-10)}) \qquad (11)$$

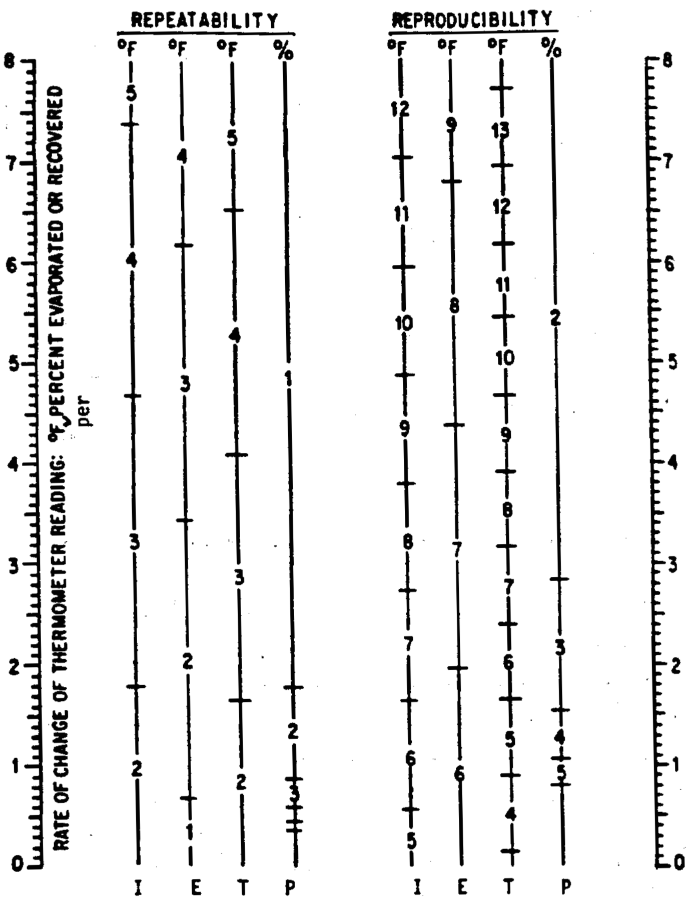
11.4.6 90 % recovered is calculated as follows:

$$C/V\%,\ F/V\% = 0.1(T_{90} - T_{80}) \qquad (12)$$

11.4.7 95 % recovered is calculated as follows:

$$C/V\%,\ F/V\% = 0.2(T_{95} - T_{90}) \qquad (13)$$

where:



NOTE—*I* = initial boiling point, °F,
E = end point (final boiling point) or dry point, °F,
T = thermometer reading at prescribed percent evaporated or recovered, °F, and
P = percent evaporated or recovered at prescribed thermometer reading, °F.

FIG. 9 Groups 2, 3, 4—Manual Method-Fahrenheit Precision of Distillation Test Method D 86 - IP 123

$C/V\%$ = rate of change in temperature at the volume percent in question, °C,
$F/V\%$ = rate of change in temperature at the volume percent in question, °F, and
T = temperature at the percent volume recovered indicated by the subscript, °C or °F,

subscripts:

V = volume percent recovered in question,
$V - 10$ = 10 % less than volume percent in question,
$V + 10$ = 10 % more than volume percent in question, and
IBP, 5, 10, 80, 90, 95 = appropriate volume percent indicated.

11.5 *Bias:*

11.5.1 *Absolute Bias*—Due to the use of total immersion thermometers or temperature sensing systems designed to emulate them, the distillation temperatures in this test method are somewhat lower than the true temperature. The amount of absolute bias has not been determined.

11.5.2 *Relative Bias*—There exists a bias between the empirical results of distillation properties obtained by this

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test method and the true boiling point distillation curve obtained by Test Method D 2892. The amount of relative bias between the two test methods has not been determined.

11.5.2.1 *Groups 1, 2, 3, and 4*—Refer to Tables A1.2 A1.3, and A1.4 for the statement of bias between automatic and manual apparatus.

12. Keywords

12.1 distillates; distillation; petroleum products

TABLE 10 Observed Precision Values for Typical Values of Slope Groups 2, 3, and 4 (Automatic)

% Collected	Slope		Repeatability		Reproducibility	
	°C/ %V	°F/ %V	°C	°F	°C	°F
20 to 70 %	0.5	0.9	1.5	2.7	4.5	8.1
	1.0	1.8	2.5	4.5	6.5	11.7
	1.5	2.7	3.0	5.4	8.5	15.3
	2.0	3.6	4.0	7.2	10.5	18.9
	2.5	4.5	4.5	8.1	12.5	22.5
10 and 80 %	0.5	0.9	1.5	2.7	4.0	7.2
	1.0	1.8	2.5	4.5	5.5	9.9
	1.5	2.7	3.0	5.4	7.0	12.6
	2.0	3.6	4.0	7.2	8.0	14.4
	2.5	4.5	4.5	8.1	9.5	17.1
5, 90, and 95 %	1.0	1.8	2.0	3.6	4.5	8.1
	2.0	3.6	3.0	5.4	7.0	12.6
	3.0	5.4	4.0	7.2	9.5	17.1
	4.0	7.2	5.0	9.0	12.0	21.6
2 %	2.0	3.6	3.5	6.3	6.0	10.8
	3.0	5.4	3.5	6.3	8.0	14.4
	4.0	7.2	3.5	6.3	10.0	18.0
	5.0	9.0	3.5	6.3	12.0	21.6
IBP	...	...	3.5	6.3	8.5	15.3
FBP	...	...	3.5	6.3	10.5	18.9

ANNEX

(Mandatory Information)

TABLE A1.1 Summary of Aids for Definition of Repeatability and Reproducibility

Group	Distillation Method	Temperature Scale	Table, Section, and Figure to Use
0	Manual or Automatic	°C or °F	11.2.1
1	Manual	°C	Table 7 and Fig. 4
		°F	Table 7 and Fig. 5
1	Automatic	°C	Table 8 and Fig. 6
		°F	Table 8 and Fig. 7
2,3,4	Manual	°C	Fig. 8
		°F	Fig. 9
2,3,4	Automatic	°C	Tables 9 or 10
		°F	Tables 9 or 10

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TABLE A1.2 Condensed Summary of Comparative Manual and Automatic Distillation Results

NOTE—All thermometer readings were corrected to 101.3 kPa (760 mm Hg) pressure. The left-hand figures were manually obtained, and the right-hand figures represent corresponding results from the automatic apparatus.

Celsius					
Gasoline—Twenty six laboratories and fourteen samples (see Table A1.3)					
Kerosine—Eight tests in four laboratories for manual, and six tests in three laboratories for automatic					
Max	176.5, 174.5	193.5, 193	215.5, 215.5	248, 248.5	268, 268.5
Min	171.5, 172	191, 190.5	213.5, 214	245.5, 246.5	264, 265
Average	174.5, 173.5	191.5, 191.5	214.5, 214.5	246, 247	265.5, 266.5
Diesel Fuel—Ten tests in five laboratories for manual and for automatic					
Max	190.5, 189	215, 218	268.5, 269	322, 323	341.5, 343
Min	179.5, 179.5	208.5, 208.5	264, 264	318, 316	337, 338.5
Average	185.5, 184.4	213, 214	266, 266	319.5, 318.5	340, 340,5
Fahrenheit					
Gasoline—Twenty six laboratories and fourteen samples (See Table A1.4)					
	Initial Boiling Point	10 % Evaporated	50 % Evaporated	90 % Evaporated	End Point (Final Boiling Point)
Kerosine—Eight tests in four laboratories for manual, and six tests in three laboratories for automatic					
Max	350, 346	380, 379	420, 420	478, 479	514, 515
Min	341, 342	376, 375	416, 417	474, 476	507, 509
Average	346, 344	377, 377	418, 418	475, 477	510, 512
Diesel Fuels Ten tests in five laboratories for manual and for automatic					
Max	375, 372	419, 424	515, 516	612, 613	647, 649
Min	355, 355	407, 407	507, 507	604, 601	639, 641
Average	366, 364	415, 417	511, 511	607, 605	644, 645

TABLE A1.3 Bias Between Methods (ADA-Manual) °C (Based on Averages of ASTM and IP Data)

Sample	IBP	5 %	10 %	20 %	30 %	40 %	50 %	60 %	70 %	80 %	90 %	95 %	FBP	Sample
1	+1.1	+1.9	+2.2	+1.6	+1.4	+0.7	+0.8	+0.7	+0.7	+0.1	+0.4	+0.7	−0.4	1
2	(+0.9)	(0.0)	+0.8	+0.5	+0.4	+0.6	+0.2	+0.1	+0.1	+0.4	(+4.7)[B]	(+1.3)[B]	(−1.2)[B]	2
3	+0.7	+1.4	+1.6	+1.0	+0.8	+0.6	+0.3	+0.1	+0.2	+0.9	+0.5	+0.1	−0.8	3
4	+0.3	+0.6	+0.8	+0.8	+0.3	+0.7	+0.6	+0.8	+1.1	+1.2	+0.8	+0.5	−0.9	4
5	+0.5	+1.3	+1.3	+1.3	+1.2	+1.0	+0.9	+0.6	+0.8	+1.0	+0.4	+0.4	−0.9	5
6	+1.2	+1.2	+1.6	+1.2	+1.2	+1.1	+0.8	+1.1	+1.2	+0.2	−0.1	+0.2	−0.3	6
7	+0.3	+0.8	+0.8	+0.7	+0.8	+0.8	+1.0	+1.5	+1.6	+1.6	+1.5	+1.7	−0.7	7
8	+0.3	+0.5	+0.7	+0.6	+0.7	+1.2	+1.2	+1.1	+1.3	+1.9	+1.1	+1.2	−0.8	8
9	+1.7	+2.0	+1.8	+1.5	+1.5	+1.5	+1.2	+0.9	+1.3	+0.6	−0.4	+0.4	−1.2	9
10	+1.5	+1.5	+1.2	+0.7	+0.4	+0.6	+0.9	+1.0	+1.4	+1.9	+0.9	+0.1	−2.1	10
11	+0.9	+1.1	+1.2	+0.8	+0.7	+0.6	+1.1	+1.0	+0.4	+0.5	−0.4	+0.1	−0.8	11
12	+1.0	(+2.4)[B]	+2.3	+1.2	+1.2	+1.2	+1.2	+0.9	+1.1	+0.2	−0.7	(−0.8)[B]	−0.9	12
13[A]	+0.3	+0.3	+0.4	+0.3	+0.2	+0.9	+1.4	+1.0	+0.1	+1.1	+1.2	+1.0	−1.2	13[A]
14[A]	+0.5	+0.4	+0.7	+0.5	+0.8	+1.1	+1.7	+1.7	+1.0	+0.8	+0.3	0.0	−0.8	14[A]

[A] Gasohols.

[B] ( ) Points not included in the precision analysis.

TABLE A1.4 Bias Between Methods (ADA-Manual) °F (Based on Averages of ASTM and IP Data)

Sample	IBP	5 %	10 %	20 %	30 %	40 %	50 %	60 %	70 %	80 %	90 %	95 %	FBP	Sample
1	+1.9	+3.4	+4.0	+2.9	+2.5	+1.2	+1.4	+1.2	+1.3	+0.1	+0.8	+1.2	−0.7	1
2	(+1.6)[B]	(0.0)[B]	+1.5	+0.9	+0.7	+1.1	+0.4	+0.2	+0.1	+0.7	(+8.4)[B]	(+2.3)[B]	(−2.2)[B]	2
3	+1.2	+2.5	+2.8	+1.7	+1.4	+1.0	+0.5	+0.1	+0.4	+1.6	+0.9	+0.1	−1.4	3
4	+0.5	+1.1	+1.5	+1.4	+0.6	+1.2	+1.1	+1.4	+1.9	+2.1	+1.4	+0.9	−1.7	4
5	+0.9	+2.3	+2.3	+2.3	+2.1	+1.7	+1.6	+1.0	+1.4	+1.8	+0.7	+0.8	−1.7	5
6	+2.1	+2.2	+2.8	+2.2	+2.1	+2.0	+1.4	+2.0	+2.1	+0.4	−0.1	+0.4	−0.5	6
7	+0.6	+1.4	+1.4	+1.3	+1.5	+1.5	+1.8	+2.6	+2.8	+2.8	+2.6	+3.1	−1.3	7
8	+0.6	+0.9	+1.3	+1.0	+1.2	+2.1	+2.1	+2.0	+2.4	+3.4	+2.0	+2.1	−1.4	8
9	+3.1	+3.5	+3.2	+2.7	+2.6	+2.7	+2.1	+1.6	+2.3	+1.1	−0.7	+0.8	−2.1	9
10	+2.7	+2.7	+2.1	+1.2	+0.8	+1.0	+1.6	+1.7	+2.5	+3.4	+1.6	+0.2	−3.8	10
11	+1.6	+2.0	+2.1	+1.4	+1.2	+1.0	+1.9	+1.7	+0.8	+0.9	−0.7	+0.2	−1.4	11
12	+1.8	(+4.2)[B]	+4.1	+2.1	+2.2	+2.1	+2.1	+1.6	+1.9	+0.4	−1.2	(−1.5)[B]	−1.7	12
13[A]	+0.5	+0.5	+0.7	+0.5	+0.3	+1.6	+2.5	+1.7	+0.2	+2.0	+2.1	+1.8	−2.2	13[B]
14[A]	+0.9	+0.7	+1.2	+0.9	+1.4	+2.0	+3.0	+3.0	+1.8	+1.5	+0.6	0.0	−1.4	14[B]

[A] Gasohols.

[B] ( ) Points not included in the precision analysis.

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APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES ILLUSTRATING CALCULATIONS FOR REPORTING OF DATA

X1.1 *Thermometer Readings Corrected to 101.3 kPa (760 mm Hg) Pressure:*

X1.1.1 *Thermometer Readings Correction to 101.3 kPa (see 10.3):*

$$\text{correction (°C)} = 0.0009\,(101.3 - 98.6)\,(273 + t_c) \quad \text{(X1.1)}$$

$$\text{correction (°F)} = 0.00012\,(760 - 740)\,(460 + t_f). \quad \text{(X1.2)}$$

X1.1.2 *Loss Correction to 101.3 kPa (see 10.5):*

$$\text{corrected loss} = (0.750 \times 4.7) + 0.125 = 3.6. \quad \text{(X1.3)}$$

X1.1.3 *Recovery Correction to 101.3 kPa (see 10.5.2):*

$$\text{corrected recovery} = 94.2 + (4.7 - 3.6) = 95.3. \quad \text{(X1.4)}$$

X1.2 *Thermometer Readings at Prescribed Percentages Evaporated:*

X1.2.1 *Thermometer Reading at 10 % Evaporated (5.3 % Recovered) (see 10.8.1):*

$$T_{10E}(\text{°C}) = 33.5 + [(40.5 - 33.5)(5.3 - 5)/(10 - 5)] = 33.9\text{°C} \quad \text{(X1.5)}$$

$$T_{10E}(\text{°F}) = 92 + [(104 - 92)(5.3 - 5)/(10 - 5)] = 92.7\text{°F} \quad \text{(X1.6)}$$

X1.2.2 *Thermometer Reading at 50 % Evaporated (45.3 % Recovered) (See 10.8.1):*

$$T_{50E}(\text{°C}) = 94 + [(109 - 94)(45.3 - 40)/(50 - 40)] = 102.0\text{°C} \quad \text{(X1.7)}$$

$$T_{50E}(\text{°F}) = 201 + [(228 - 201)(45.3 - 40)/(50 - 40)] = 215.3\text{°F} \quad \text{(X1.8)}$$

X1.2.3 *Thermometer Reading at 90 % Evaporated (85.3 % Recovered) (See 10.8.1):*

$$T_{90E}(\text{°C}) = 181.5 + [(201.0 - 181.5)(85.3 - 85)/(90 - 85)] = 182.7\text{°C} \quad \text{(X1.9)}$$

$$T_{90E}(\text{°F}) = 359 + [(394 - 359)(85.3 - 85)/(90 - 85)] = 361.1\text{°F} \quad \text{(X1.10)}$$

X1.2.4 *Thermometer Reading at 90 % Evaporated (85.3 % Recovered) Not Corrected to 101.3 kPa Pressure (See 10.8.1):*

$$T_{90E}(\text{°C}) = 180.5 + [(200.4 - 180.5)(85.3 - 85)/(90 - 85)] = 181.7\text{°C} \quad \text{(X1.11)}$$

$$T_{90E}(\text{°F}) = 357 + [(392 - 357)(85.3 - 85)/(90 - 85)] = 359.1\text{°F} \quad \text{(X1.12)}$$

TABLE X1.1 Thermometer Readings Corrected to 101.3 kPa (760 mm Hg) Pressure

Barometric Pressure	Observed 98.6 kPa (740 mm Hg)		Corrected 101.3 kPa (760 mm Hg)	
	°C	°F	°C	°F
Initial Boiling Point	25.5	78	26.0	79
5 % recovered	33.0	91	33.5	92
10 % recovered	39.5	103	40.0	104
15 % recovered	46.0	115	46.5	116
20 % recovered	54.5	130	55.0	131
30 % recovered	74.0	165	75.0	167
40 % recovered	93.0	199	94.0	201
50 % recovered	108.0	226	109.0	228
60 % recovered	123.0	253	124.0	255
70 % recovered	142.0	288	143.5	290
80 % recovered	166.5	332	168.0	334
85 % recovered	180.5	357	181.5	359
90 % recovered	200.4	392	201.0	394
95 % recovered	...	...	...	...
End Point	215.0	419	216	421
Recovery, %	94.2	...	95.3	...
Residue, %	1.1	...	1.1	...
Loss, %	4.7	...	3.6	...

ATTACHMENT 9

X2. CALCULATION OF REPEATABILITY AND REPRODUCIBILITY OF VOLUME % (RECOVERED OR EVAPORATED) AT A PRESCRIBED TEMPERATURE READING

X2.1 Some specifications require the reporting of the volume % evaporated or recovered at a prescribed temperature. The statistical determination of the precision of the volume % evaporated or recovered at a prescribed temperature has not been directly measured for Groups 0 or 1 in an interlaboratory program. The following procedure makes the assumption that the precision of the volume % evaporated or recovered at a prescribed temperature is equivalent to the precision of the temperature measurement at that point divided by the rate of change of temperature versus volume % evaporated or recovered. The estimation becomes less valid at high slope values.

X2.2 Calculate the rate of change in thermometer reading in degrees Celsius (or Fahrenheit) per the volume % evaporated or recovered ($°C(F)/V\%$) in accordance with 11.4 using temperature values which are higher and lower than the prescribed temperature in question.

X2.3 Calculate the repeatability (r_o) or reproducibility of temperature (R_o) or both of the temperature using the value of $°C(F)/V\%$ as determined in X2.2 and the appropriate table or figure as indicated in Table A1.1.

X2.4 Determine the repeatability or reproducibility or both of the volume % evaporated or recovered at a prescribed temperature from the following formulas:

$$^{r}\text{vol}\,\% = r_o/(°C(F)/V\%) \qquad ^{R}\text{vol}\,\% = R_o/(°C(F)/V\%) \tag{X2.1}$$

where:

^{r}vol % = repeatability of the volume % evaporated or recovered,
^{R}vol % = reproducibility of the volume % evaporated or recovered,
r_o = repeatability of the temperature at the prescribed temperature,
R_o = reproducibility of the temperature at the prescribed temperature, and
$°C(F)/V\%$ = rate of change in thermometer reading in degrees Celsius (Fahrenheit) per the volume % evaporated or recovered.

TABLE X2.1 Distillation Data From a Group 1 Sample Manual Distillation

Distillation Point Recovered, mL	Temperature, °C	Temperature, °F	Volume (mL) Recovered at 93.3 (200) °C (F)
			18.0
10	84	183	
20	94	202	
30	103	217	
40	112	233	

Distillation Point Evaporated, mL	Temperature, °C	Temperature, °F	Volume (mL) Evaporated at 93.3 (200) °C (F)
			18.4
10	83	182	
20	94	201	
30	103	217	
40	111	232	

X2.5 *Example Calculation:*

X2.5.1 For a Group 1 sample exhibiting distillation characteristics as per Table X2.1. To determine ^{r}vol % evaporated at 93.3°C (200°F) (from 11.4.5).

$°C/V\%, F/V\% = 0.05\,(T_{(v+10)} - T_{(v-10)})$
$°C/V\% = 0.05\,(T_{(30)} - T_{(10)})$
$°C/V\% = 0.05\,(103 - 83)$
$°C/V\% = 0.05\,(20)$
$°C/V\% = 1.0$

$°C/V\%, F/V\% = 0.05\,(T_{(v+10)} - T_{(v-10)})$
$°F/V\% = 0.05\,(T_{(30)} - T_{(10)})$
$°F/V\% = 0.05\,(217 - 182)$
$°F/V\% = 0.05\,(35)$
$°F/V\% = 1.75$

X2.5.2 From Fig. 4 (°C) or Fig. 5 (°F):

$r_o = 0.864\,(°C/V\%) + 1.214$
$r_o = 0.864\,(1.0) + 1.214$
$r_o = 2.078$

$r_o = 0.864\,(°F/V\%) + 2.186$
$r_o = 0.864\,(1.75) + 2.186$
$r_o = 3.698$

X2.5.3 From X2.4:

$^{r}\text{vol}\,\% = r_o/(°C/V\%)$
$^{r}\text{vol}\,\% = 2.078/1.0$
$^{r}\text{vol}\,\% = 2.1$

$^{r}\text{vol}\,\% = r_o/(°F/V\%)$
$^{r}\text{vol}\,\% = 3.698/1.75$
$^{r}\text{vol}\,\% = 2.1$

ATTACHMENT 9