Designation: D 1078 – 01

Designation: 195/98

Standard Test Method for Distillation Range of Volatile Organic Liquids[1]

This standard is issued under the fixed designation D 1078; 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.

1. Scope *

1.1 This test method covers the determination of the distillation range of liquids boiling between 30 and 350°C, that are chemically stable during the distillation process, by manual or automatic distillation procedures.

1.2 This test method is applicable to organic liquids such as hydrocarbons, oxygenated compounds, chemical intermediates, and blends thereof.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

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.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure[2]

E 1 Specification for ASTM Thermometers[3]

E 133 Specification for Distillation Equipment[4]

E 299 Test Method for Trace Amounts of Peroxides in Organic Solvents[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *initial boiling point*—the temperature indicated by the distillation thermometer at the instant the first drop of condensate leaves the condenser tube.

3.1.2 *dry point*—the temperature indicated at the instant the last drop of liquid evaporates from the lowest point in the distillation flask, disregarding any liquid on the side of the flask.

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *final boiling point*—the maximum thermometer reading obtained during the test.

3.2.1.1 *Discussion*—This usually occurs after the evaporation of all liquid from the bottom of the flask. The term "maximum temperature" is a frequently used synonym.

3.2.2 *end point 5 minutes*—the thermometer reading obtained 5 min after the 95 % distillation point if no dry or final boiling point occurs.

4. Summary of Test Method

4.1 A 100-mL specimen is distilled under conditions equivalent to a simple batch differential distillation. The temperature of the mercury in the thermometer is equilibrated with that of the refluxing liquid before the distillate is taken over. Boiling temperatures observed on a partial immersion thermometer are corrected to standard atmospheric pressure to give true boiling temperatures.

5. Significance and Use

5.1 This test method provides a method of measurement of distillation range of volatile organic liquids. The relative volatility of organic liquids can be used with other tests for identification and measurement of quality. Therefore, this test method provides a test procedure for assessing compliance with a specification.

5.2 This test method also provides an empirical value of residue, solvent recovery capacity, and loss (or non-recovery) on heating. Organic liquids are used as solvents in many chemical processes. As the relative volatility, residual matter and recovery capability affect the efficiency of these processes, this test method is useful in manufacturing control.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

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

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

[5] *Annual Book of ASTM Standards*, Vol 15.05.

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

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6. Apparatus

6.1 *Distillation Apparatus*—See Condenser and Cooling Bath section, Figs. 1 and Figs. 2, and Metal Shield or Enclosure for Flask section of Specification E 133.

6.2 *Distillation Flasks*, 200-mL of borosilicate glass complying with the specifications given in Distillation Flask section, Fig. 3, and Flask C of Specification E 133.

NOTE 1—Liquid superheating in a new flask may be prevented by depositing a small amount of carbon in the bottom of the flask. This may be accomplished by heating and decomposing a pinch of tartaric acid in the bottom of the flask. The flask is then prepared for use by washing with water, rinsing with acetone, and drying.

6.3 *Source of Heat*—An adjustable gas burner or electric heater so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in Section 8. For narrow-range (less than 2°C) liquids, an electric heater may be used only if it has been proven to give results comparable to those obtained when using gas heat. (See Section 9 for factors that cause superheating, and Appendix X1 for a discussion on the use of electric heaters.)

6.4 *Distillation Receiver*:

6.4.1 *Manual Distillation Receiver*—A 100-mL cylinder graduated in 1 mL subdivision and having an overall height of 250 to 260 mm.

6.4.2 *Automatic Distillation Receiver*—A receiver to be used with automatic distillation in accordance with the instrument manufacturer's instructions, conforming to the dimensions given in 6.4.1, with the exception of the graduations.

6.4.2.1 *Automatic Distillation Level Follower*—The level follower/recording mechanism of the automatic apparatus shall have a resolution of 0.1 mL with an accuracy of ± 1 mL.

6.5 *Temperature Measurement Devices*:

6.5.1 *Manual Distillation Thermometers*—Partial immersion thermometers as listed in Table 1, conforming to Specification E 1. Both bore corrections and either ice or steam standardization corrections are recommended.

6.5.2 *Automatic Distillation Temperature Sensors*—Temperature measurement systems using thermocouples or resistance thermometers shall exhibit the same temperature lag and accuracy as the appropriate and calibrated mercury-in-glass thermometer. Confirmation of the calibration of these temperature sensors shall be done at regular intervals. This can be accomplished potentiometrically by the use of standard precision resistance, depending on the type of probe. Another technique is to distill pure toluene (99.9 + % purity) and compare the temperature indicated by the thermocouple or resistance thermometer with that shown by the mercury-in-glass thermometer.

NOTE 2—Toluene is shown in reference manuals as boiling at 110.6°C (corrected for barometric pressure) under the conditions of a manual D 1078 distillation that uses a partial immersion thermometer.

TABLE 1 Thermometers

ASTM Thermometer Number	IP	Range, °C	Subdivision, °C
2C[A]	62C[A]	−5 to + 300	1.0
3C[A]	73C[A]	−5 to + 400	[B]
14C	...	38 to 82	0.1
37C	77C	−2 to + 52	0.2
38C	78C	24 to 78	0.2
39C	79C	48 to 102	0.2
40C	80C	72 to 126	0.2
41C	81C	98 to 152	0.2
42C[A]	82C[A]	95 to 255	0.5
102C	83C	123 to 177	0.2
103C	84C	148 to 202	0.2
104C	85C	173 to 227	0.2
105C	86C	198 to 252	0.2
106C	87C	223 to 277	0.2
107C	88C	248 to 302	0.2

[A] These thermometers have more temperature lag than the other thermometers listed herein and are not satisfactory for use with narrow-boiling range liquids.
[B] 1 to 301°C; 1.5°C above 301°C.

7. Hazards

7.1 **Precaution**—Certain solvents and chemical intermediates, particularly, but not only ethers and unsaturated compounds, may form peroxides during storage. These peroxides may present a violent explosion hazard when the chemical is distilled, especially as the *dry point* is approached. When peroxide formation is likely because of chemical type or length of storage, the material should be analyzed for peroxides (See Test Method E 299.) and if they exist in hazardous concentrations, appropriate precautions should be taken such as destroying the peroxide before distillation, shielding, or destroying the sample and not running the test.

7.2 Most organic solvents and chemical intermediates will burn. In the operation of the distillation apparatus, use a suitable catch pan and shielding to contain spilled liquid in the event of accidental breakage of the distillation flask.

7.3 Provide adequate ventilation to maintain solvent vapor concentrations below the lower explosive limit in the immediate vicinity of the distillation apparatus, and below the threshold limit value in the general work area.

8. Preparation of Apparatus

8.1 *Manual Distillation Apparatus*:

8.1.1 Clean and dry the condenser tube by swabbing with a piece of soft lint-free cloth attached to a wire or cord, or by any other suitable means.

8.1.2 Use the thermometer listed in the material specification for the product under study. If no thermometer is specified, select one from Table 1 with the smallest graduations that will cover the entire distillation range of the material. Center the thermometer into the neck of the flask through a tight-fitting cork stopper so that the upper end of the contraction chamber (or bulb if Thermometer 2C or IP thermometer 62C is used) is level with the lower side of the vapor tube at its junction with the neck of the flask. (See Fig. 1 of Test Method D 86.)

NOTE 3—It is far more important that the greatest volume of mercury be immersed in the refluxing zone than that the immersion mark on the thermometer be placed at any specific point.

8.1.3 Position the correct heat shield (see 9.1.3.1 and 9.1.3.2).

NOTE 4—For low-boiling materials, cool the apparatus to room temperature before starting the test.

8.1.4 Fill the condenser bath with water of the appropriate temperature shown in Table 2.

NOTE 5—When distilling pure compounds always ensure that the

TABLE 2 Temperatures

Initial Boiling Point,° C	Condenser, °C	Sample, °C
Below 50	0 to 3	0 to 3
50 to 70	0 to 10	10 to 20
70 to 150	25 to 30	20 to 30
Above 150	35 to 50	20 to 30

condenser bath temperature is above the crystallizing point of the compound.

8.1.5 Adjust the temperature of the appropriate portion of the sample to the applicable temperature shown in Table 2.

8.2 *Automatic Distillation Apparatus*—For assembly of automatic distillation apparatus, consult instrument manufacturer's operating manual.

9. Procedure

9.1 *Manual Distillation Procedure*:

9.1.1 Using the graduated receiver measure 100 ± 0.5 mL of the temperature-adjusted sample. Remove the flask from the apparatus and transfer the fresh specimen directly to the flask, allowing the graduate to drain for 15 to 20 s.

NOTE 6—For viscous liquids, a longer drainage period may be necessary to complete the transfer of the specimen to the flask, but the drainage time should not exceed 5 min. Do not allow any of the specimen to enter the vapor tube.

9.1.2 Connect the flask to the condenser by inserting the vapor tube of the flask into the condenser, making a tight connection with a well-rolled cork or similar material. Adjust the position of the heat shield so that the neck of the flask is vertical and the vapor tube extends into the condenser tube a distance of 25 to 50 mm. Have the bottom of the flask resting firmly in the hole of the heat shield. Insert the thermometer as described in 8.1.2. Place the receiver, without drying, at the outlet of the condenser tube in such a position that the condenser tube extends into the graduate at least 25 mm but does not extend below the 100-mL mark. If the initial boiling point of the material is below 70°C, immerse the cylinder in a transparent bath and maintain at a temperature of 10 to 20°C throughout the distillation. Place a flat cover on the top of the graduate to prevent condensed moisture from entering the graduate.

9.1.3 A certain amount of judgment is necessary in choosing the best operating conditions to get acceptable accuracy and precision for materials having different distilling temperatures. As a general guide, it is recommended that:

9.1.3.1 For materials having an initial boiling point below 150°C, the following conditions be established:

(a) (*a*) *Heat Shield*—Hole size, 32-mm diameter.

(b) (*b*) *Heating Rate*—Time from application of heat to first drop of distillate, 5 to 10 min, and time of rise of vapor column in neck of flask to side arm, 2½ to 3½min.

9.1.3.2 For materials having an initial boiling point above 150°C, the following conditions should be established:

(a) (*a*) *Heat Shield*—Hole size, 38-mm diameter.

(b) (*b*) *Heating Rate*—Time from application of heat to first drop of distillate, 10 to 15 min, and time of rise of vapor column in neck of flask to side arm, sufficiently rapid to permit collection of the first drop of distillate within 15 min of the start of heating.

9.1.4 Adjust the heat input so that the distillation proceeds at a rate of 4 to 5 mL/min (approximately 2 drops per second), and move the receiving cylinder so that the tip of the condenser tube touches one side of the cylinder after the first drop falls (initial boiling point). Record the readings of the distillation thermometer after collecting 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 95 mL of distillate.

9.1.5 Without changing the heater setting, continue distillation beyond the 95 % point until the dry point is observed. Record the temperature at this point as the dry point (Section 3). If a dry point is not obtained (that is, if active decomposition should occur before the dry point is reached, as evidenced by a rapid evolution of vapor or heavy fumes; or if there is liquid remaining on the bottom of the flask when the maximum temperature is observed on the distillation thermometer), record this fact.

9.1.6 When a dry point cannot be obtained, report as the end point the maximum temperature observed on the distillation thermometer or final boiling point (Section 3). When active decomposition is encountered, the rapid evolution of vapor and heavy fumes is usually followed by a gradual decrease in the distillation temperature. Record the temperature and report as the decomposition point (Section 3). If the expected drop in temperature does not occur, record the maximum temperature observed on the distillation thermometer 5 min after the 95 % point has been reached, and report as "end point, 5 min." This notation shows that a true end point could not be reached within the given time limit. In any event, the end point should not exceed 5 min after the 95 % point.

9.1.7 Read and record the barometric pressure.

9.1.8 After the condenser tube has drained, read the total volume of distillate and record it as recovery. The total yield of distillate from a material having a distillation range of 10°C or less should be not less than 97 % for nonviscous liquids. For viscous liquids and materials having a wider distillation range than 10°C, a yield of 95 volume % is satisfactory. If yields are not obtained within these limits, repeat the test.

9.1.9 If any residue is present, cool to room temperature and pour into a small cylinder graduated in 0.1-mL subdivisions. Measure the volume and record it as residue. Record the difference between 100 and the sum of the residue plus recovery as distillation loss.

9.2 *Automatic Distillation Procedure*:

9.2.1 Using the automatic distillation receiver measure 100 ± 0.5 mL of the temperature-adjusted sample. Transfer the fresh specimen directly to the flask, allowing the receiver to drain for 15 to 20 s (see Note 6).

9.2.2 Connect the distillation flask to the condenser and fit the temperature measuring device to the flask according to the instrument manufacturer's instruction.

9.2.3 Start the distillation following the instrument manufacturer's instruction.

10. Factors Causing Superheating

10.1 In general, any condition whereby the temperature surrounding the vapor exceeds the temperature of the vapor in equilibrium with the liquid will cause superheating. Specific

factors conducive to superheating are as follows, and should be avoided:

10.2 *Flame in Contact with the Flask*— The applied gas flame should be prevented from contacting more than the specified portion of the flask by the following procedures:

10.2.1 Maintain the correct overall dimensions and specified hole diameter of the asbestos cement board. The hole must be perfectly circular, with no irregularities.

10.2.2 Use a board that is free of cracks and checks.

10.2.3 Set the flask snugly in the hole in the upper insulating board.

10.3 *Application of Heat*—Attention should be given to burner placement, position, and character of flame, as follows:

10.3.1 Apply the source of heat directly beneath the flask. Any variation would result in heating a larger portion of surrounding air to a higher temperature than that of the flask.

10.3.2 The flame should not have a larger cross section than is necessary, and should be nonluminous.

10.3.3 Place the burner at a level such that the complete combustion area of a nonluminous flame is approximately ¾ in. (20 mm) below the board.

10.4 *Extraneous Heat Source*—An extraneous source of heat such as sunlight falling directly on the flask can cause superheating.

10.5 *Condition of Equipment*—Observe caution in employing the apparatus for immediate reuse. For low-boiling materials, cool the heating unit to room temperature before starting the test.

10.6 *Use of Electric Heaters*—Electric heaters generally cause superheating. These should be used only after they have been proven to give results comparable to those obtained when using gas heat. The superheating effect obtained from electric heaters may be minimized, but not completely eliminated, by selecting a heater that, by its design, concentrates the heating elements to a minimum area, and contains a minimum amount of ceramic material in its overall construction. The fulfillment of these requirements will reduce, but not completely eliminate, the amount of extraneous heat radiating around the perimeter of the asbestos-cement board on which the distillation flask is placed.[6] (See Appendix X1 for a more complete discussion of the problems encountered in the use of electric heaters.)

11. Calculations

11.1 *Manual Distillation Calculations*:

11.1.1 *Thermometer Bore Correction*—Apply the corrections for any variations in the bore of the thermometer as given by the calibration.

11.1.2 *Thermometer Bulb Shrinkage Correction*—Apply the correction for shrinkage of the mercury bulb of the thermometer as determined by any change in its ice or steam point where applicable. Other means can be employed, such as the use of a platinum-resistance thermometer or a National Bureau of Standards thermometer.

11.1.3 *Barometer Pressure Correction*—After applying the corrections for thermometer error, correct each reading for deviation of the barometric pressure from normal by adding algebraically the correction calculated as follows:

11.1.3.1 For values of K in degrees Celsius per millimetre of mercury

$$\text{Correction} = K(760 - P) \quad (1)$$

where:

K = rate of change of boiling point with pressure, in degrees Celsius per millimetre, as given in Table 3 (Note 7), and

P = barometric pressure in millimetres of mercury at standard temperature.

11.1.3.2 For values of K in degrees Celsius per millibar:

TABLE 3 Change of Boiling Point with Pressure

Compound	Value of K, °C per mm Hg at Boiling Point	K, °C per mbar at Boiling Point	Boiling Point at 760 mm Hg, °C
Acetone	0.039	0.029	56.1
n-Amyl alcohol	0.041	0.031	138.0
n-Amyl acetate	0.048	0.036	149.5
Aromatic solvent naphtha	0.049	0.037	...
Benzene	0.043	0.032	80.1
Isobutyl acetate	0.045	0.035	117.3
n-Butyl acetate	0.045	0.035	126.1
sec-Butyl acetate	0.045	0.034	112.4
Isobutyl alcohol	0.036	0.027	107.9
n-Butyl alcohol	0.037	0.028	117.7
sec-Butyl alcohol	0.035	0.026	99.5
Diacetone alcohol	0.050	0.037	...
Diethylene glycol	0.050	0.037	245.0
Dipropylene glycol	0.051	0.038	232.8
Ethyl acetate	0.041	0.030	77.2
Ethyl alcohol	0.033	0.025	78.3
Ethylene glycol	0.045	0.033	197.6
2-Butoxyethanol	0.047	0.035	171.2
2-Ethoxyethanol	0.044	0.033	135.1
2-Ethoxyethyl acetate	0.046	0.035	156.3
Hexylene glycol	0.045	0.033	197.1
n-Hexyl acetate	0.050	0.037	171.6
Isophorone	0.057	0.043	215.3
Methyl alcohol	0.033	0.025	64.5
Methyl ethyl ketone	0.043	0.032	79.6
Methyl isoamyl acetate	0.048	0.036	146.2
Methyl isoamyl ketone	0.048	0.036	144.9
Methyl isobutyl carbinol	0.041	0.030	131.8
Methyl isobutyl ketone	0.046	0.035	116.2
Perchloroethylene	0.048	0.036	121.2
Isopropyl alcohol	0.033	0.025	82.3
Isopropyl acetate	0.041	0.030	88.5
Propylene glycol	0.043	0.032	187.6
Pyridine	0.046	0.035	115.4
Toluene	0.046	0.035	110.6
Trichloroethylene	0.043	0.032	87.1
Vinyl acetate	0.040	0.030	72.7
Xylene (mixed isomers)	0.049	0.037	...

[6] The sole source of supply of the Lo-Cap heater known to the committee at this time is Precision Scientific Co., Chicago, IL. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

$$\text{Correction} = K(1013 - P) \quad (2)$$

where:

K = rate of change of boiling point with pressure, in degrees Celsius per millibar, as given in Table 3 (Note 7), and

P = barometric pressure in millibars at standard temperature and pressure.

NOTE 7—For other pure compounds not listed in Table 3, the value K should be obtained from the literature. For narrow-boiling hydrocarbon materials, the value of K may be assumed as 0.00012 times the normal boiling point on the absolute temperature scale.

11.1.4 If the overall distillation range of the sample does not exceed 2°C, combined thermometer (bore irregularities and bulb shrinkage) and barometric corrections may be made on the basis of the difference between the observed 50 % boiling point and the true boiling point at 760 mm as given in Table 3.

11.2 *Automatic Distillation Calculations*—Barometric correction shall be done according to 11.1.3 and 11.1.4.

12. Report

12.1 Report the results in a manner conforming with the specifications of the material tested. If no definite manner of reporting is specified, report the corrected temperatures at each observed volume, and report the volume percentages of residue, recovery, and distillation loss.

13. Precision and Bias

13.1 *Interlaboratory Study*[7]:

13.2 The precision of this test method was obtained from an interlaboratory study conducted in 2000 involving manual and automatic distillation procedures. The study involved six samples of different boiling point ranges, done in duplicate. Ten laboratories did automatic D 1078 distillation, and five laboratories did manual D 1078 distillation. It was found that the precision is dependent on the boiling point temperature. The data were statistically evaluated using the D2PP software available from ASTM.

13.3 *Repeatability*—Two results, each the mean of two runs, obtained by the same operator should be considered suspect if they differ by more than the repeatability values shown in Tables 4 and 5 at a 95 % confidence level.

13.4 *Reproducibility*—Two results, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than the reproducibility values shown in Tables 4 and 5 at a 95 % confidence level.

[7] Supporting data are available from ASTM Headquarters. Request RR: D01-1123.

TABLE 5 Sample Calculation of D 1078 Precision Values, °C

Manual D 1078 RR—IBP Data			Auto D 1078 RR—IBP Data		
IBP	*r*	*R*	IBP	*r*	*R*
50	0.3	0.5	50	0.2	0.8
75	0.4	0.8	75	0.3	1.2
100	0.5	1.1	100	0.5	1.6
125	0.7	1.3	125	0.6	2.0
150	0.8	1.6	150	0.7	2.3
175	0.9	1.9	175	0.8	2.7
Manual D 1078 RR—50 % Data			**Auto D 1078 RR—50 % Data**		
50 %	*r*	*R*	50 %	*r*	*R*
50	0.2	0.3	50	0.1	0.3
75	0.4	0.5	75	0.2	0.5
100	0.5	0.6	100	0.2	0.7
125	0.6	0.8	125	0.3	0.9
150	0.7	1.0	150	0.3	1.0
175	0.8	1.1	175	0.4	1.2
Manual D 1078 RR—DP Data			**Auto D 1078 RR—DP Data**		
DP	*r*	*R*	DP	*r*	*R*
50	0.3	0.7	50	0.2	0.5
75	0.5	1.0	75	0.3	0.8
100	0.7	1.3	100	0.4	1.1
125	0.9	1.6	125	0.5	1.3
150	1.0	2.0	150	0.6	1.6
175	1.2	2.3	175	0.7	1.9
Manual D 1078 RR—BPR[A] Data			**Auto D 1078 RR—BPR Data**		
BPR	*r*	*R*	BPR	*r*	*R*
0.5	0.2	0.6	0.5	0.2	0.7
5	0.4	1.2	5	0.4	1.3
10	0.6	1.7	10	0.7	1.9
15	0.8	2.3	15	0.9	2.6
20	1.0	2.9	20	1.1	3.2
25	1.2	3.5	25	1.3	3.9
30	1.4	4.1	30	1.6	4.5

[A] BPR = boiling point range.

13.5 *Bias*:

13.5.1 *Absolute Bias*—Since the temperature measuring devices specified by this test method are calibrated against the normal boiling point of toluene (99.9+ % purity), this test method has no bias with respect to pure toluene as a reference material.

13.5.2 *Relative Bias Between Manual and Automatic D 1078 Distillation*—Statistical comparison between the variances of automatic and manual D 1078 distillation results did not indicate any statistically significant difference. Statistical comparison of the averages of the six samples used in the study indicated that the paired-sample, two-tailed, t-test for the initial boiling point (IBP) and 50 % distillation point showed a small relative bias that is not statistically significant. A small but statistically significant bias was indicated for the automatic and manual D 1078 dry point (DP). Table 6 gives a summary of the relative bias observed for the samples used in the study. The observed bias (if any) are only for the samples studied and may not be necessarily applicable to other samples.

TABLE 4 Summary of D 1078 Precision Data, °C

Parameter	Manual D 1078		Automatic D 1078	
	Repeatability, *r*	Reproducibility, *R*	Repeatability, *r*	Reproducibility, *R*
IBP	0.00528 *X*	0.0107 *X*	0.00450 *X*	0.0156 *X*
50 %	0.00471 *X*	0.00647 *X*	0.00209 *X*	0.00683 *X*
DP	0.00684 *X*	0.01301 *X*	0.00396 *X*	0.01070 *X*
Boiling range	0.0405 (*X* + 5)	0.1163 (*X* + 5)	0.0445 (*X* + 5)	0.1294 (*X* + 5)

where X = the mean of two results being compared.

TABLE 6 Relative Bias (Auto–Manual) for D 1078 Distillation, °C

Sample	Initial Boiling Point		50 % B.P.		Dry Point	
	Auto	Manual	Auto	Manual	Auto	Manual
Toluene	110.19	110.16	110.61	110.54	110.67	110.71
Commercial nonene	136.31	136.07	137.72	137.68	142.49	142.25
Special cut naphtha	167.99	167.25	170.98	170.16	179.05	178.63
Light naphtha	98.26	98.05	101.66	101.65	114.76	114.03
Naphtha	118.47	118.24	126.03	125.59	144.73	144.02
Mineral spirits	162.13	160.40	172.71	171.65	193.11	192.71
	Relative Bias		Relative Bias		Relative Bias	
Toluene	0.03		0.07		−0.04	
Commercial nonene	0.24		0.04		0.24	
Special cut naphtha	0.74		0.82		0.42	
Light naphtha	0.21		0.01		0.73	
Naphtha	0.23		0.44		0.71	
Mineral spirits	1.73		1.06		0.40	
Average relative bias	0.53		0.41		0.41	
F–test (95 % confidence)	Equivalent		Equivalent		Equivalent	
t–test (95 % confidence)	Equivalent		Equivalent		Not equivalent	

NOTE 8—In cases of dispute, the parties involved may agree to designate either the manual or the automatic method to be the referee test method. If an agreement on which method to designate cannot be made, the referee test method will be the manual method.

14. Keywords

14.1 distillation range; solvents

APPENDIX

(Nonmandatory Information)

X1. DISCUSSION ON THE USE OF ELECTRIC HEATERS WHEN APPLYING TEST METHOD D1078/IP 195 TO THE DETERMINATION OF THE DISTILLATION RANGE OF NARROW RANGE (<2°C) PURE COMPOUNDS

X1.1 Test Method D 1078/IP 195, in the hands of a competent operator using properly designed equipment, has been found over the years to be a valuable tool in detecting the presence of low-boiling and high-boiling impurities in relatively pure compounds.

X1.2 In recent years many laboratories, for reasons of safety and convenience, have eliminated the availability of natural or artifical gas, with the resulting trend toward the exclusive use of electric heaters in place of gas burners. The use of electricity instead of gas as the source of heat, coupled with the application of this test method to materials of extremely high purity and narrow distillation ranges (2°C or less), has resulted in the distortion of the dry point. This distortion effect can be illustrated by comparing the distillation range results using gas and electric heat on a sample of high-purity methanol (Table X1.1). The purity of the methanol employed was established by gas chromatography and other instrumental procedures.

X1.3 The higher dry point obtained with the electric heater is due to the large amount of extraneous heat radiating around the distillation flask which in turn is due to the relatively large area of the distillation board exposed to the heating elements of the electric heater. For example, the heating elements of the electric heater[5] cover an area of 63.5 by 76 mm whereas the flame of a properly adjusted gas burner can be concentrated to an area no larger than the 32-mm hole of the supporting asbestos cement (or ceramic) board. This means that when the heating elements of the electric heater are brought up to sufficient temperature to effect the proper distillation rate of the methanol, a relatively large area of the board also is exposed to the heat of these elements so that as the end of the distillation is approached, a considerable amount of heat is being radiated from the board to the air surrounding the distillation flask. This hot air surrounding the flask is sufficient to cause a distortion of the dry point as the last drop of liquid is vaporized from the bottom of the flask. Conversely, the ability to concentrate the gas flame to only the 32-mm exposed area of the flask minimizes the extent of the extraneous heat radiating from the board, which, in turn, eliminates the distortion of the dry point from this cause. This conclusion was substantiated by results from the following experiment: The board with the 32-mm

TABLE X1.1 Comparison of Gas Versus Electric Heat

	Gas Heat		Electric Heater[5]	
		Range		Range
Determination 1:				
Initial boiling point	64.5	0.3	64.5	1.5
Dry point	64.8		66.0	
Determination 2:				
Initial boiling point	64.5	0.4	64.5	1.9
Dry point	64.9		66.4	
Mean range	...	0.35	...	1.7

hole was replaced by a 190.7 by 190.7-mm stainless steel plate in which a 32-mm hole had been cut. Four turns of 6.4-mm copper tubing with sufficient inlet and outlet leads were silver-soldered to the underside of the plate so that water could be circulated through the tubing during the course of the distillation. With this "water-cooled board" substituted for the standard board, only the heat from the electrical elements immediately under the 32-mm outlet could reach the distillation flask. The heat emanating from the outer perimeter of the heater was dissipated by the water circulating through the tubing on the underside of the board, as evidenced by the ability to hold one's finger on top of the" water-cooled board" during the course of the distillation. Using the same high-purity methanol employed in the previous experiment, distillation ranges were determined using gas heat, electric heat, and electric heat with the "water-cooled board" substituted for the standard board. These results are given in Table X1.1.

X1.4 The results from the above experiment demonstrate the effect the extraneous radiant heat from the electric heater had on the dry point, and also suggest a means whereby this effect could be greatly reduced.

X1.5 Despite the fact that the use of a "water-cooled board" in conjunction with an electric heater eliminated the extraneous radiant heat surrounding the distillation flask, it is evident from the results in Table X1.2 that the dry point obtained when using the electric heater and "water-cooled board" was still significantly higher than the dry point obtained when using gas heat. The assumption was made that this "residual" interference to the dry point was caused by infrared radiation from the glowing electric heating elements. This assumption has been supported by the following experimental evidence: The bottom of a standard 200-mL distillation flask was coated with a 38-mm diameter circle of black ceramic marking ink. The black ink was in turn fired into the glass by heating the bottom of the flask to a dull-red heat with a gas-oxygen glass blowtorch. The presence of this black coating on the bottom of the flask would absorb any infrared radiation emanating from the electrical heating elements, thus preventing it from affecting the bulb of the thermometer at the end of the distillation. Distillation ranges were then determined on the same methanol as used in the previous experiments following Test Method D 1078/IP 195, except that in one case a standard 200-mL flask was used with an electric heater plus the "water-cooled board," and in the other case, the specially prepared "black bottom" 200-mL flask was employed. Both of these special conditions were compared to the standard procedure using gas heat. The results of this experiment, given in Table X1.3, show that the use of the "black-bottom flask" in conjunction with the electric heater and "water-cooled board" causes a significant lowering of the dry point which confirms the theory that infrared radiation emanating from the electric heating elements causes a slight distortion of the dry point.

X1.6 The above discussion and experimental evidence are presented to show how and why the use of electric heater causes a distortion of the dry point when carrying out the procedure as specified in Test Method D 1078/IP 195. Although this distortion is of a minor nature, and therefore of little importance when applying this test method to compounds or mixtures which have distillation ranges of 5°C or more, the effect becomes significant when the method is applied to narrow range (2°C or less) pure compounds. It is the further objective of this discussion to suggest to those laboratories equipped with only electricity, a technique whereby the distillation range results obtained with electric heaters may be made equivalent to those obtained with gas heat.

X1.7 The sponsoring subcommittee of this test method has not had the opportunity to apply this technique to a sufficiently large number of compounds over a wide range of boiling temperatures to warrant including it as a part of Test Method D 1078/IP 195; and is, therefore, presenting it for information purposes only. Comments are solicited from those attempting to employ the suggestions contained in this report as well as other ideas that might be employed to equate the use of electric heat with gas heat.

TABLE X1.2 Comparison of Electric Heat Plus Water-Cooled Board to Gas Heat

	Gas Heat		Electric Heat[5]		Electric Heat[5] plus Water-Cooled Board	
		Range		Range		Range
Determination 1:						
Initial boiling point	64.5	0.3	64.5	1.6	64.5	0.5
Dry point	64.8		66.1		65.0	
Determination 2:						
Initial boiling point	64.6	0.3	64.5	1.4	64.5	0.6
Dry point	64.9		65.9		65.1	
Mean range	...	0.3	...	1.5	...	0.55

TABLE X1.3 Effect of Infrared Radiation on Dry Point

	Gas Heat		Electric Heater[5] with Water-Cooled Board and Standard Flask		Electric Heater[5] with Water-Cooled Board and Black-Bottom Flash	
		Range		Range		Range
Determination 1:						
Initial boiling point	64.5	0.3	64.5	0.6	64.6	0.2
Dry point	64.8		65.1		64.8	
Determination 2:						
Initial boiling point	64.5	0.4	64.6	0.6	64.6	0.2
Dry point	64.9		65.2		64.9	
Mean range	...	0.35	...	0.6	...	0.25

SUMMARY OF CHANGES

Committee D1 has identified the location of selected changes to this standard since the last date of issue that may impact the use of this standard.

(*1*) A new Section 13 replaced the old Section 13 to incorporate new precision values for automatic and manual D 1078 distillation, obtained from a 2000 interlaboratory study.
(*2*) A new Table 4 replaced the old Table 4.
(*3*) A new Table 5 replaced the old Table 5.
(*4*) A new Table 6 has been added.
(*5*) Footnote 7 was modified and Footnote 8 was deleted.

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