



Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure (Micro Distillation Method)¹

This standard is issued under the fixed designation D 7345; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers a procedure for determination of the distillation characteristics of petroleum products having boiling range between 20 to 400°C at atmospheric pressure using an automatic micro distillation apparatus.

1.2 This test method is applicable to such products as; light and middle distillates, automotive spark-ignition engine fuels, automotive spark-ignition engine fuels containing up to 10 % ethanol, aviation gasolines, aviation turbine fuels, regular and low sulfur diesel fuels, biodiesel fuels, blends up to 20 % biodiesel, special petroleum spirits, naphthas, white spirits, kerosines, burner fuels, and marine fuels.

1.3 The test method is also applicable to hydrocarbons with a narrow boiling range, like organic solvents or oxygenated compounds.

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

1.5

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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 All standards are subject to revision, and parties to agreement on this test method are to apply the most recent edition of the standards indicated below, unless otherwise specified, such as in contractual agreements or regulatory rules where earlier versions of the method(s) identified may be required.

2.2 ASTM Standards:²

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

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

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)

D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)

D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)

D 5482 Test Method for Vapor Pressure of Petroleum Products (Mini Method Atmospheric)

D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

D 6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D 6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

2.3 Energy Institute Standards:³

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

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

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

IP 69 Petroleum products - Determination of vapour pressure - Reid method

IP 394 Liquid petroleum products - Vapour pressure - Part 1: Determination of air saturated vapour pressure (ASVP)

2.4 ISO Standards:⁴

Guide 34 General requirements for the competence of reference material producers

Guide 35 Reference materials — General and statistical principles for certification

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automatic apparatus, n*—microprocessor-controlled unit that performs the procedures of automatically controlling the evaporation of a liquid specimen under specific conditions of this test method, collecting measurement data and converting this data by patented algorithm in order to predict distillation results in correlation with industry recognized reference method.

3.1.2 *corrected temperature reading, n*— temperature readings, as described in 3.1.12, corrected to 101.3 kPa barometric pressure.

3.1.3 *end point (EP) or final boiling point (FBP), n*—maximum corrected temperature readings obtained during the test at the instant the flask internal pressure returns to the initial pressure level registered by automatic apparatus.

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

3.1.4 *flask internal pressure, n*—pressure within the distillation flask obtained during the test by a differential pressure sensor of automatic apparatus.

3.1.4.1 *Discussion*—The flask internal pressure data recorded during the test is automatically converted to the volume percent recovered or evaporated data by patented algorithm employed by automatic apparatus.

3.1.5 *initial boiling point (IBP), n*—corrected temperature readings that corresponds to the instant of the flask internal pressure rise registered by automatic apparatus.

3.1.6 *liquid temperature, n*—temperature of the liquid specimen in the distillation flask during the test obtained by a liquid temperature measuring device of automatic apparatus.

3.1.7 *percent evaporated, n*—percent recovered corrected to a predicted by automatic analyzer evaporation loss percent. Percent evaporated is automatically reported for ASTM 7C thermometer correlation.

3.1.8 *percent recovered, n*—volume percent automatically reported by the analyzer; expressed as a percentage of the charge volume, associated with a simultaneous temperature readings. Percent recovered is reported for ASTM 8C thermometer correlation.

3.1.9 *percent recovery, n*—percent recovery predicted by the automatic apparatus and expressed as a percentage of the charge volume.

3.1.10 *percent residue, n*—volume of residue in the distillation flask predicted by the automatic apparatus and expressed as a percentage of the charge volume.

3.1.11 *reference method, n*—ASTM D 86 test method or its analog which is widely used for expression of the distillation characteristics of petroleum products in industry.

3.1.12 *temperature readings, n*—vapor and liquid temperature has through use of an algorithm of the automatic apparatus been adjusted to mimic the same temperature lag and emergent stem effects as would be seen when using an ASTM 7C/7F or 8C/8F liquid-in-glass thermometer to determine the distillation characteristics of the material under test by industry recognized reference method.

3.1.13 *vapor temperature, n*—temperature of the vapors in the distillation flask during the test obtained by a vapor temperature measuring device of automatic apparatus.

4. Summary of Test Method

4.1 A specimen of the sample is transferred into the distillation flask, the distillation flask is placed into position on the automatic apparatus, and heat is applied to the bottom of the distillation flask.

4.2 The automatic apparatus measures and records specimen vapor and liquid temperatures, and pressure in the distillation flask as the sample gradually distills under atmospheric pressure conditions. Automatic recordings are made throughout the distillation and the data stored into the apparatus memory.

4.3 At the conclusion of the distillation, the collected data is treated by the data processing system, converted to distillation characteristics and corrected for barometric pressure.

4.4 Test results are commonly expressed as percent recovered or evaporated versus corresponding temperature in compliance with industry recognized standard form and reference method either in a table or graphically, as a plot of the distillation curve.

5. Significance and Use

5.1 The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the

⁴ Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.2 The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up, 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.

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

5.4 This test method can be applied to contaminated products or hydrocarbon mixtures. This is valuable for fast product quality screening, refining process monitoring, fuel adulteration control, or other purposes including use as a portable apparatus for field testing.

5.5 This test method uses an automatic micro distillation apparatus, provides fast results using small sample volume, and eliminates much of the operator time and subjectivity in comparison to Test Method D 86.

6. Apparatus

6.1 *Basic Components of the Automatic Apparatus:*⁵

6.1.1 The basic components of the micro distillation unit are the distillation flask, a condensate recovery area with waste beaker, an enclosure for the distillation flask with the heat source and flask support, the specimen liquid temperature measuring device, the specimen vapor temperature measuring device, the distillation flask internal pressure measuring device, the ambient pressure measuring device, the control systems for regulating the distillation process, and the data processing system for converting recorded information into typical industry recognized standard report form.

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

6.3 *Barometer for Calibration*—A pressure measuring device capable of measuring local station pressure with an accuracy of 0.1 kPa (1 mmHg) or better, at the same elevation relative to sea level where the apparatus is located.

6.3.1 The barometer is only required for periodic calibration of the external and internal pressure measuring devices.

6.3.2 (**Warning**—Do not take readings from ordinary aneroid barometers, such as those used at weather stations and airports, since these are precorrected to give sea level readings.)

6.4 *Sampling Device*—Glass or plastics syringe capacity 10 ± 0.3 mL or constant volume dispenser capacity 10 ± 0.3 mL.

6.5 *Waste Beaker*—Glass approximately 200 mL capacity, outside diameter approximately 70 mm and height approximately 130 mm fitted with a cover to reduce evaporation. The cover design shall allow the beaker to remain open to atmospheric pressure.

7. Reagents and Materials

7.1 *Cleaning Solvents*, suitable for cleaning and drying the test flask such as; petroleum naphtha and acetone. (**Warning**—Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.)

7.2 *Toluene*, 99.5 % purity. (**Warning**—Extremely flammable. Harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard.)

7.3 *n-Hexadecane*, 99 % purity. (**Warning**—Extremely flammable. Harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard.)

7.4 Chemicals of at least 99 % purity shall be used in the calibration procedure (see 10.2). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁶

7.5 *Granular Pumice Stones*, clean and dry fine grade pumice stones of diameter 0.8 to 3.0 mm, approximately 10 grains are necessary for each test.

7.6 *Sample Drying Agent*—Anhydrous sodium sulfate has been found to be suitable.

8. Sampling, Storage, and Sample Conditioning

8.1 *Sampling:*

8.1.1 The extreme sensitivity of volatility measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution in the drawing and handling of volatile product samples.

8.1.2 Obtain a sample and test specimen in accordance with Practice D 4057, D 4177 or D 5854 when appropriate. At least 50 mL of sample is recommended.

8.1.3 Sample shall be free from any suspended solids or other insoluble contaminations. Obtain another sample or remove solid particle by filtration. During filtration operation take care to minimize any loss of light ends.

⁵ The sole source of supply of the apparatus known to the committee at this time is ISL /PAC, B.P. 70285 Verson, 14653 CARPIQUET – FRANCE. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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

8.2 Sample Storage:

8.2.1 All samples shall be stored in a tightly closed and leak-free container away from direct sunlight or sources of direct heat.

8.2.2 Protect samples containing light materials having expected initial boiling point lower than 100°C (212°F) from excessive temperatures prior to testing. This can be accomplished by storage of the sample container in an appropriate ice bath or refrigerator at a temperature below 10°C. Other samples can be stored at ambient or lower temperature.

8.2.3 If the sample has partially or completely solidified during storage, it is to be carefully heated to a temperature when it is completely fluid. It shall be vigorously shaken after melting, prior to opening the sample container, to ensure homogeneity.

8.3 Wet Samples:

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

8.3.2 If such a sample cannot be obtained, remove any free water by placing approximately 30 mL of the sample to be tested in a glass conical flask containing approximately 10 g of the drying agent. Stopper and shake gently. Allow the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample for the analysis. It is recommended to filter the test portion from the residual or suspended drying agent. During this drying and filtration operations take care to minimize any loss of light ends. Report that the sample has been dried by the addition of a drying agent.

9. Preparation of Apparatus

9.1 Install the analyzer for operation in accordance with the manufacturer's instructions.

9.2 This instrument shall be located away from direct sunlight, sources of direct heat or air draft.

9.3 Turn on the main power switch of the analyzer.

10. Verification Quality Control Checks

10.1 To verify the temperature measurement system, distill high purity toluene in accordance with this test method and comparing the temperature reading at 50 % distilled. If the temperature reading differs more than 0.5°C from the expected temperature of 109.3°C (see Note 1), then check the instrument calibration (see Section 11).

10.2 To verify the temperature measurement system at elevated temperatures, use *n*-hexadecane and record the temperature at 50 % distilled. If the temperature reading differs more than 1.0°C from the expected temperature of 278.6°C (see Note 1, then check the instrument calibration (see Section 11).

NOTE 1—These temperatures are those that would be obtained if the toluene and hexadecane were distilled using Test Method D 86 and are not the figures that are given as the boiling points of these materials in literature.

10.3 Verification of apparatus performance under dynamic conditions and wide temperature range can be done by distillation of a Certified Reference Material (CRM) or Secondary Working Standard (SWS).

10.3.1 *Certified Reference Material (CRM)*— CRM is a stable mixture of hydrocarbon or other stable petroleum product with a method-specific distillation characteristic established by a method-specific interlaboratory study following Practice D 6300 or ISO Guide 34 and Guide 35. Suppliers of CRMs will provide certificates stating the method-specific distillation characteristic for each material of the current production batch.

10.3.2 *Secondary Working Standard (SWS)*— SWS is a stable mixture of pure hydrocarbons, or other petroleum product whose composition is known to remain appreciably stable. Establish the mean value of control points and the statistical control limits for the SWS using standard statistical techniques. See Practice D 6299.

11. Calibration

11.1 Follow the manufacturer's instructions for verifying the correct operation of the apparatus.

11.2 *Temperature Measurement System*— At intervals of not more than six months or after the system has been replaced or repaired, or both, following the apparatus instruction manual, check the calibration of the liquid and vapor temperature measuring sensors by distilling of pure compounds, like toluene and *n*-hexadecane.

NOTE 2—The melting point of *n*-hexadecane is 18°C. If the sample is solid, heat it to about 25°C and wait until all the material is liquid before starting the test.

11.3 *External Pressure Measuring Device*— At intervals of not more than six months, or after the system has been replaced or repaired, or both, the external pressure measuring device reading of the apparatus shall be verified against a barometer, as described in 6.3.

11.4 *Differential Pressure Measuring Device*—At intervals of not more than six months, or after the system has been replaced or repaired, or both, the differential pressure measuring device reading of the apparatus shall be verified in accordance with the manufacturer's instructions.

12. Procedure

12.1 ~~Material with an initial boiling point~~ Initial Boiling Point of 100°C (212°F) and below: Bring Below—Bring the temperature of the sample and container to a temperature at least 10°C below the expected initial boiling point of the material before opening the sample container.

12.2 ~~Material with a boiling point~~ **Boiling Point above 100°C (212°F):** ~~Bring~~—Bring the sample and its container to ambient temperature. If the sample has partially or completely solidified during storage warm until fluid, then mix by gentle shaking.

NOTE 3—Not respecting the requirements given in 12.1 can lead to improper IBP detection on samples containing volatile materials. If expected initial boiling point of the sample to be tested is unknown, it is advised to make a test at ambient conditions. If the distillation result shows that the requirements of 12.1 were not respected, discard the result and repeat the test strictly respecting the conditions.

12.3 Ensure that the distillation measuring head of automatic apparatus has been allowed to reach ambient temperature and that any residual condensate has been removed.

12.4 Check that the distillation flask is clean and dry.

12.5 Place at least 10 grains of clean and dry granular pumice stones into the distillation flask. Some apparatus supply a suitable boiling stone dispenser.

12.6 Measure 10 ± 0.3 mL test portion using the sampling device (see Section 6). Check for the presence of any bubbles and if present discard the test portion and refill with bubble free material. Transfer the bubble free test portion to the prepared distillation flask, taking care that none of the liquid flows into the vapor tube.

NOTE 4—Use new disposable syringe or disposable dispenser tip for each new sampling to avoid any products cross contamination which can cause erroneous distillation results.

12.7 Fit the distillation measurement head into its position on the distillation flask securely in accordance with the manufacturer's instructions.

12.8 Place the distillation flask into the heating enclosure and insert the sidearm of the distillation flask into the sealing of the condenser tube, while also attaching the measurement head holder.

12.9 Position the heating source/flask support around the lower section of the distillation flask.

12.10 Close heating enclosure by positioning the protection shield to its position.

12.11 Check that a waste collection beaker is placed under the projecting lower end of the condenser tube. Ensure that the waste collection beaker remains open to atmospheric pressure.

NOTE 5—Monitor that the liquid level in the waste beaker does not exceed two thirds of its total capacity and drain it on periodic intervals.

12.12 Without delay initiate the distillation process according to the apparatus manufacturer's instructions.

12.12.1 From this point up to and including the termination of the measurement, the apparatus automatically controls all operations. The instrument applies heating to the specimen and regulates automatically heating power during the distillation run using specimen liquid temperature data. The distillation conditions; distillation flask internal pressure, specimen liquid temperature, and specimen vapor temperature are automatically measured and recorded during the test by the control system. The distillation is automatically terminated when the flask internal pressure returns to its initial pressure level. The collected test data is automatically processed and reported on the display and printed out at the conclusion of the test run. The heating enclosure cooling fan is automatically activated.

12.13 Record the test data.

12.14 Allow the distillation flask to cool and remove it from the apparatus.

13. Report

13.1 In cases in which no specific data requirements have been set by the operator, corrected temperatures readings versus corresponding percent recovered or evaporated are typically reported by the apparatus. Report typically contains the IBP, FBP, 5 %, 95 % and each 10 % increment from 10 to 90 %, as well as percent recovery and percent residue.

13.1.1 If required, the percent loss is calculated by the following equation:

$$\text{loss \%} = 100 - (\text{percent recovery} + \text{percent residue}) \quad (1)$$

13.2 Report all volumetric percentages to the nearest 0.1 % (V/V).

13.3 Report all temperature readings to the nearest 0.1°C (see Note 3).

13.4 Report if a drying agent, as described in 8.3.2, was used.

13.5 The test report shall contain at least the following information:

13.5.1 Sufficient details for complete identification of the product tested;

13.5.2 A reference to this standard;

13.5.3 The result of the test;

13.5.4 Any deviation, by agreement or otherwise, from the procedure specified; and

13.5.5 The date of the test.

14. Precision and Bias

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory⁷ test results is as follows:

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1621.

NOTE 6—Typically samples for distillation are classified according to a Group number (see Test Method D 86). However, this test method does not require this classification, but for the purposes of precision and cross method reproducibility comparisons, the precisions and bias have been derived according to the group number in the following fashion. Group 1, 2, and 3 samples are labeled as NOT4, and Group 4 samples are labeled GRP4. See Appendix X1 for further information on typical samples and group classification.

NOTE 7—Information on the precision of % evaporated or % recovered at a prescribed temperature can be found in Annex A4.

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

Group NOT4: Refer to Annex A2 for charts of calculated repeatability.

Group NOT4: Refer to Annex A2 for tables of calculated repeatability.

IBP:	$r = E^{1.464} \times 0.01$	valid range: 20 – 80°C
E10:	$r = (E + 192.3) \times 0.0055$	valid range: 35 – 95°C
E50:	$r = 1.54$	valid range: 60 – 140°C
E90:	$r = 1.31$	valid range: 110 – 245°C
FBP:	$r = 4.42$	valid range: 140 – 260°C

Group GRP4: Refer to Annex A2 for charts of calculated repeatability.

Group GRP4: Refer to Annex A2 for tables of calculated repeatability.

IBP:	$r = 3.9$	valid range: 145 – 195°C
T10:	$r = T \times 0.00954$	valid range: 160 – 265°C
T50:	$r = T \times 0.00614$	valid range: 170 – 295°C
T90:	$r = T \times 0.0041$	valid range: 180 – 340°C
T95:	$r = 2.01$	valid range: 260 – 338°C
FBP:	$r = 3.93$	valid range: 195 – 365°C

where:

E = evaporated temperature within valid range prescribed

T = recovered temperature within valid range prescribed

NOTE 7—For 8—For naphthas, solvents, and other similar materials where percent recovered is reported and the percent loss is typically less than one percent, the percent recovered temperatures can be considered identical to the percent evaporated temperatures, and precision can be calculated as shown for Group NOT4.

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

Group NOT4: Refer to Annex A2 for charts of calculated repeatability.

Group NOT4: Refer to Annex A2 for tables of calculated repeatability.

IBP:	$R = E^{1.464} \times 0.01595$	valid range: 20 – 80°C
E10:	$R = (E + 192.3) \times 0.01$	valid range: 35 – 95°C
E50:	$R = 2.64$	valid range: 60 – 140°C
E90:	$R = 2.82$	valid range: 110 – 245°C
FBP:	$R = 8.1$	valid range: 140 – 260°C

Group GRP4: Refer to Annex A2 for charts of calculated repeatability.

Group GRP4: Refer to Annex A2 for tables of calculated repeatability.

IBP:	$R = 6.0$	valid range: 145 – 195°C
T10:	$R = T \times 0.0177$	valid range: 160 – 265°C
T50:	$R = T \times 0.0103$	valid range: 170 – 295°C
T90:	$R = T \times 0.0081$	valid range: 180 – 340°C
T95:	$R = 3.28$	valid range: 260 – 338°C
FBP:	$R = 7.7$	valid range: 195 – 365°C

where:

E = evaporated temperature within valid range prescribed

T = recovered temperature within valid range prescribed

NOTE 8—See Note 7 9—See Note 8.

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

14.3 *Relative Bias*—The Degree of Agreement between results by Test Method D 7345 and Test Method D 86 (automated)—Results on the same materials produced by Test Method D 7345 and Test Method D 86 have been assessed in accordance with procedures outlined in Practice D 6708.⁷ The findings are:

IBP:

NOT4

Bias-corrected results from Test Method D 7345, as per the bias correction equation (Eq 2), may be considered as practically equivalent to results from Test Method D 86 (automated), for sample types and property ranges studied. No sample-specific bias, as defined in Practice D 6708, was observed after the bias-correction, for the materials studied.

$$\text{Bias-corrected } X = \text{predicted } Y = 0.87 X + 5.88^\circ\text{C} \quad (2)$$

where:

X = result obtained by Test Method D 7345 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 2 and Test Method D 86, for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

No bias-correction considered in Practice D 6708 can further improve agreement between results from Test Method D 7345 and Test Method D 86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D 6708, was observed for some samples.

Differences between results from Test Method D 7345 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T10:

NOT4

No bias-correction considered in Practice D 6708 can further improve the agreement between results from Test Method D 7345 and Test Method D 86 (automated), for sample types and property ranges studied. Sample-specific bias, as defined in Practice D 6708, was observed for some samples.

Differences between results from Test Method D 7345 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

The degree of agreement between results from Test Method D 7345 and Test Method D 86 (automated) can be further improved by applying the bias-correction outlined in Eq 3. Sample-specific bias, as defined in Practice D 6708, was observed for some samples after applying the bias-correction.

$$\text{Bias-corrected } X = \text{predicted } Y = 1.09 X - 16.4^{\circ}\text{C} \quad (3)$$

where:

X = result obtained by Test Method D 7345 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 3 and Test Method D 86, for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T50:

NOT4

The degree of agreement between results from Test Method D 7345 and Test Method D 86 (automated) can be further improved by applying the bias-correction outlined in Eq 4. Sample-specific bias, as defined in Practice D 6708, was observed for some samples after applying the bias-correction.

$$\text{Bias-corrected } X = \text{predicted } Y = X - 1.37^{\circ}\text{C} \quad (4)$$

where:

X = result obtained by Test Method D 7345 (this test method), and *bias-corrected* X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 4 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

Note—Users are cautioned to verify Eq 4 for winter grade RFG (10 % Ethanol), since this material was not included in the above assessment.

GRP4

The degree of agreement between results from Test Method D 7345 and Test Method D 86 (automated) can be further improved by applying the bias-correction outlined in Eq 5. Sample-specific bias, as defined in Practice D 6708, was observed for some samples after applying the bias-correction.

$$\text{Bias-corrected } X = \text{predicted } Y = X - 2.015^{\circ}\text{C} \quad (5)$$

where:

X = result obtained by Test Method D 7345 (this test method), and bias-corrected X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 5 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

T90:

NOT4

The degree of agreement between results from Test Method D 7345 and Test Method D 86 (automated) can be further improved by applying the bias-correction outlined in Eq 6. Sample-specific bias, as defined in Practice D 6708, was observed for some samples after applying the bias-correction.

$$\text{Bias-corrected } X = \text{predicted } Y = 0.98 X + 1.76^{\circ}\text{C} \quad (6)$$

where:

X = result obtained by Test Method D 7345 (this test method), and bias-corrected X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 6 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

Note—Users are cautioned to verify Eq 6 for RFG (10 % Ethanol), since this material type was not included in the above assessment.

GRP4

The degree of agreement between results from Test Method D 7345 and Test Method D 86 (automated) can be further improved by applying the bias-correction outlined in Eq 7. Sample-specific bias, as defined in Practice D 6708, was observed for some samples after applying the bias-correction.

$$\text{Bias-corrected } X = \text{predicted } Y = X - 0.67^{\circ}\text{C} \quad (7)$$

where:

X = result obtained by Test Method D 7345 (this test method), and bias-corrected X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 7 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{XY}), as defined in Practice D 6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

FBP:
NOT4

Bias-corrected results from Test Method D 7345, as per the bias correction equation (Eq 8), may be considered as practically equivalent to results from Test Method D 86 (automated), for sample types and property ranges studied. No sample-specific bias, as defined in Practice D 6708, was observed after the bias-correction, for the materials studied.

$$\text{Bias-corrected } X = \text{predicted } Y = 1.03 X - 7.18^{\circ}\text{C} \quad (8)$$

where:

X = result obtained by Test Method D 7345 (this test method), and bias-corrected X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 8 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{xy}), as defined in Practice D 6708, about 5 % of the time.

Group NOT4: Refer to Annex A3.

GRP4

Bias-corrected results from Test Method D 7345, as per the bias correction equation (Eq 9), may be considered as practically equivalent to results from Test Method D 86 (automated), for sample types and property ranges studied. No sample-specific bias, as defined in Practice D 6708, was observed after the bias-correction, for the materials studied.

$$\text{Bias-corrected } X = \text{predicted } Y = 1.02 X - 5.90^{\circ}\text{C} \quad (9)$$

where:

X = result obtained by Test Method D 7345 (this test method), and bias-corrected X = predicted Y = result that would have been obtained by Test Method D 86 (automated) on the same sample.

Differences between bias-corrected results from Eq 9 and Test Method D 86 (automated), for the sample types and property ranges studied, are expected to exceed the following cross method reproducibility (R_{xy}), as defined in Practice D 6708, about 5 % of the time.

Group GRP4: Refer to Annex A3.

14.4 The precision statements were derived according to Practice D 6300 from a 2005 interlaboratory cooperative test program.⁷ ~~Sixteen laboratories have participated and analyzed 33 sample sets comprised of various fuels with temperature range of 23 to 365°C. Information on the type of samples and their average boiling points are in the research report.⁷ Sixteen laboratories have participated and analyzed 33 sample sets comprising specification grade gasolines (some containing up to 10 % ethanol), specification grade diesel (with a B5 and B20 biodiesel), specification grade heating oil, aviation turbine fuels, aviation gasolines, marine fuels, mineral spirits, and toluene. The temperature range covered was 23 to 365°C. Information on the type of samples and their average boiling points are in the research report.⁷~~

15. Keywords

15.1 batch distillation; boiling range distribution; distillates; distillation; laboratory distillation; micro distillation; petroleum products

ANNEXES

(Mandatory Information)

A1. DETAILED DESCRIPTION OF APPARATUS

A1.1 A general schematic of the micro distillation apparatus is shown in Fig. A1.1.

A1.2 *Distillation Measurement Head*—Comprised of a snug-fitting device designed for mechanically centering and correct positioning of sensors on the distillation flask without vapor leakage. The head and distillation flask are designed in such a way that it can be installed one way without any adjustment necessary. It accommodates the vapor temperature sensor, liquid temperature sensor and has a connection for the differential pressure sensor.

A1.3 *Temperature Measuring Devices* :

A1.3.1 *Vapor Temperature Sensor*—Capable of reading to 0.1°C over the range 0 to 550°C with maximum error $\pm 0.5^{\circ}$. The

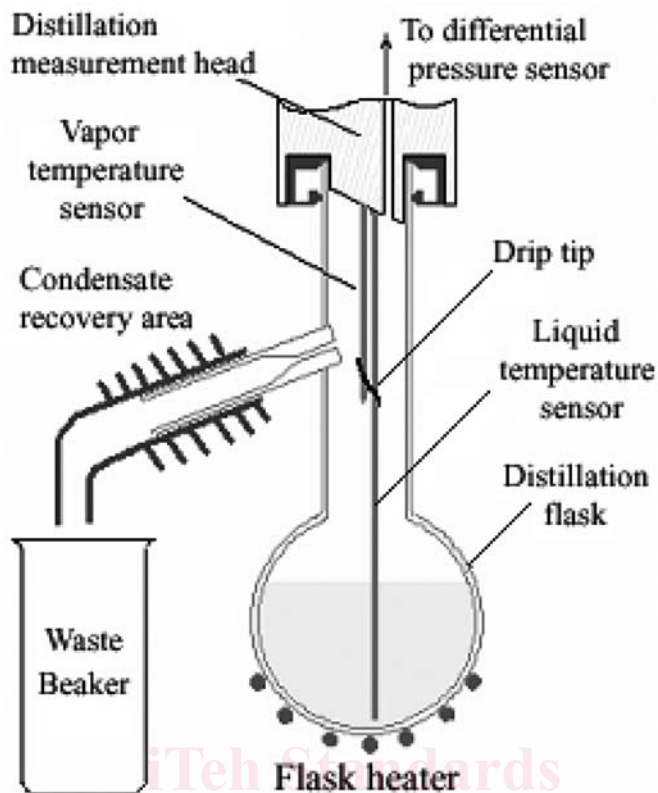


FIG. A1.1 Schematic of Micro Distillation Apparatus

bottom of the sensor shall be positioned approximately 8 mm below the sidearm opening and near the center of the distillation flask neck.

A1.3.2 *Liquid Temperature Sensor*—Capable of reading to 0.1°C over the range 0 to 550°C with maximum error $\pm 0.5^\circ$. The bottom of the sensor shall be positioned approximately 2 mm above the distillation flask bottom.

NOTE A1.1—K-type thermocouple encased in metal tube was found suitable for this purpose.

A1.4 *Drip Tip*— Special mechanical device protecting vapor temperature measurement device from excessive reflux flow.

A1.5 *Pressure Measuring Devices* :

A1.5.1 *Differential Pressure Sensor*—Comprised of a pressure line and electronic pressure transducer capable of measuring the differential pressure in the range from 0 to 2500 Pa with the resolution of 0.1 Pa and a minimum error of ± 1 Pa.

A1.5.2 *External Pressure Measuring Device*—Comprised of an electronic pressure transducer capable of measuring the ambient pressure where the apparatus is housed in the range 73.33 to 106.7 kPa with an accuracy of 0.1 kPa or better.

A1.6 *Distillation Flask*—Special micro-distillation flask made of heat resistant glass and constructed to the dimensions and tolerances shown on Fig. A1.2. The accuracy of the test method is related to distillation flask quality. Set tolerances must be respected.

A1.7 *Flask Heater*—Specially designed electric low voltage, low mass heating element positioned below the distillation flask on a movable platform and capable to supply necessary heat power and supporting the distillation flask during the test.

A1.8 *Control Systems*—Automatic controlling system capable of monitoring the collected distillation data, applying adjustments to the heating system, controlling safe and proper operation of apparatus during whole distillation run and determining the termination of the distillation.

A1.9 *Data Processing Systems* —Automatic data processing system capable of conversion of the collected distillation data into industry recognized distillation report format, correction of data for barometric pressure, with provisions for display of report or printing/transfer of report, or both.

A1.10 *Condensate Recovery Area* —Air-cooled condensate tube with provision for collection of condensed distillate and drain it to a waste beaker.