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Standard Test Method for **Distillation of Petroleum Products and Liquid Fuels at** Atmospheric Pressure (Micro Distillation Method)^{1,2}

This standard is issued under the fixed designation D7345; 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 and liquid fuels having boiling range between 20 °C 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 20 % ethanol, aviation gasolines, aviation turbine fuels, regular and low sulfur diesel fuels, biodiesel (B100), biodiesel 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.7 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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:³
- D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
- D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
- D1160 Test Method for Distillation of Petroleum Products at **Reduced Pressure**
- D4057 Practice for Manual Sampling of Petroleum and **Petroleum Products**
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D4177 Practice for Automatic Sampling of Petroleum and **Petroleum Products**
- D4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
- D5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method) (Withdrawn 2012)⁴
- D5191 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method)
- D5482 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method—Atmospheric)

¹ This test method is under the jurisdiction of ASTM International Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of ASTM Subcommittee D02.08 on Volatility. The technically equivalent standard as referenced is under the jurisdiction of the Energy Institute Subcommittee SC-B-9.

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² This test method has been developed through the cooperative effort between ASTM and the Energy Institute, London. ASTM and IP standards were approved by ASTM and EI technical committees as being technically equivalent but that does not imply both standards are identical.

³ 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.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

- D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
- 2.3 Energy Institute Standards:⁵
- 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:⁶
- ISO 17034 General Requirements for the Competence of Reference Material Producers
- Guide 35 Reference Materials—Guidance for characterization and assessment of homogeneity and stability

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *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.

3.2.2 corrected temperature reading, n— temperature readings, as described in 3.2.12, corrected to 101.3 kPa barometric pressure.

3.2.3 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.

3.2.3.1 *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.

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

3.2.4.1 *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. 3.2.5 *initial boiling point (IBP), n*—corrected temperature readings that corresponds to the instant of the flask internal pressure rise registered by automatic apparatus.

3.2.6 *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.

3.2.7 *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.

3.2.8 *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.

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

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

3.2.11 *reference method*, n—ASTM D86 test method or its analogs which is widely used for expression of the distillation characteristics of petroleum products in industry.

3.2.12 *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.

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

⁶ Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

5. Significance and Use

5.1 The distillation (volatility) characteristics of hydrocarbons and other liquids have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.2 The distillation characteristics are 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 D86.

6. Apparatus

6.1 Basic Components of the Automatic Apparatus:⁷

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 mL \pm 0.3 mL or constant volume dispenser capacity 10 mL \pm 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.⁸

7.5 *Granular Pumice Stones*, clean and dry fine grade pumice stones of diameter 0.8 mm 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 D4057, D4177, or D5854 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.

8.2 Sample Storage:

⁷ 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,¹ which you may attend.

⁸ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar 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.