ASTM D86 Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

Note

- This presentation covers those aspects of ASTM D86 which are specific to apparatus, preparation of apparatus, and calibration and standardization.
- For specific details regarding test procedure and calculations, refer to the other presentation in this training module.

Background

Scope (1.1)

- This test method addresses procedures for atmospheric distillation of petroleum products using a laboratory batch distillation unit to,
  - determine boiling range characteristics of the petroleum products quantitatively
- Petroleum products include:
  - light and middle distillates
  - automotive spark-ignition engine fuels with or without oxygenates
  - aviation gasolines
  - aviation turbine fuels

Scope (1.1)

- diesel fuels
- biodiesel blends up to 20 %
- marine fuels
- special petroleum spirits
- naphthas
- white spirits
- kerosenes
- grade 1 and 2 burner fuels

Scope (1.2, 1.3)

- The test method is designed for the analysis of distillate fuels.
- Not applicable to products containing appreciable quantities of residual material.
- Utilizes both manual and automated instruments.

Cautionary Note

Summary of Test Method (4.1)

- Sample is placed in one of four groups described in Table 1 based on,
its composition
vapor pressure
expected Initial boiling point (IBP)
expected End point (EP)

- Apparatus arrangement, condenser temperature, and other operational variables are based on the group in which the sample falls.

**Summary of Test Method (4.2)**

- 100 mL specimen of the sample is distilled under prescribed conditions for the group to which the sample belongs.
- Distillation is performed in a laboratory batch distillation unit at ambient pressure under conditions to provide approximately one theoretical plate fractionation.
- Systematic observations of temperature readings and volumes of condensate are made,
  - depending on the needs of the user of the data
- The volume of the residue and the losses are recorded.

**Summary of Test Method (4.3, 4.4)**

- At the end of the distillation, the observed vapor temperatures are corrected for barometric pressure.
- Data for conformance to procedural requirement such as distillation rates is examined.
- The test is repeated if any specified condition is not satisfied.
- The Test results are expressed as percent evaporated or percent recovered versus corresponding temperature as a plot of the distillation curve either in,
  - table
  - graph

**Significance and Use (5.1, 5.2)**

- Determine the boiling range of a petroleum product by performing a simple batch distillation.
- Distillation (volatility) characteristics of hydrocarbons are important for fuels and solvents.
- Boiling range gives information on composition, properties and 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.

**Significance and Use (5.3)**
• Distillation characteristics are important for analyzing both automotive and aviation gasolines,
  o influence engine start-up, and warm-up at low temperatures; and tendency to vapor lock at high operating temperature or at high altitude
• The presences of high boiling point components in fuels significantly affect the degree of formation of solid combustion deposits.

Significance and Use (5.4, 5.5)

• Volatility affects the rate of evaporation and is an important factor in the application of many solvents, particularly in paints.
• Distillation limits are included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

Apparatus (6)

• Basic Components
• Temperature Measuring Device
• Temperature Sensor Centering Device
• Automated Equipment
• Barometer

Basic Components (6.1)

• The basic components explained in Annex A2 is as follows.
  o distillation flasks
  o condenser and condenser bath
  o metal shield or enclosure for flask
  o heat source
  o flask support
  o flask support board
  o receiving cylinders
  o residue cylinder

Distillation Flask (A2.1)

• Made of heat resistant glass.
• Constructed according to dimensions and tolerances shown in Fig. A2.1
• Comply with the requirements of ASTM Specification E1405 or made of quartz of 99.9 % silicone dioxide.
• Can be constructed with a ground glass joint.

Condenser and Condenser Bath (A2.2, A2.2.1, A2.2.3)
• Condenser shall be made of seamless non-corrosive metal tubing,
  o 560 mm ± 5 mm in length
  o Outside diameter of 14 mm
  o Wall thickness of 0.8 mm to 0.9 mm
• Position the condenser within the condenser bath based on conditions described in Sections A2.2.2 and A2.2.3.

**Metal Shield or Enclosure for Flask (A2.3)**

• Manual units,
  o shield for gas burner
  o shield for electric heater

**Shield for Gas Burner (A2.3.1)**

• Provides protection to the operator and allows easy access to the burner and to the distillation flask during operation.
• 480 mm height.
• 280 mm long.
• 200 mm wide.
• Shall be made of sheet metal of 0.8 mm thickness.
• Has a window to observe the dry point at the end of the distillation.

**Shield for Electric Heater (A2.3.2)**

• 440 mm high.
• 200 mm long.
• 200 mm wide.
• Shall be made of sheet metal of approximately 0.8 mm thickness.
• Has a window to observe the dry point at the end of the distillation.

**Heat Source (A2.4)**

• Gas burner
• Electric heater

**Gas Burner (A2.4.1)**

• Has a sensitive manual control valve and a gas pressure regulator to give complete control of heating.
• Capable of bringing over the first drop from a cold start within the time specified, and of continuing the distillation at the specified rate.

**Electric Heater (A2.4.2)**

• Has a low heat retention characteristic.
Flask Support (A2.5)

- Type 1
- Type 2

Type 1 (A2.5.1)

- Is a flask support with a gas **burner**.
- Consists of either a ring support of the ordinary laboratory type or a platform adjustable from outside of the shield.
  - ring support - 100 mm or larger in diameter, and provides support on a stand inside the shield
- Mounted on ring or platform is a hard board made of ceramic or other heat-resistant material of,
  - 3 mm to 6 mm in thickness with a central opening 76 mm to 100 mm in diameter
  - outside line dimensions slightly smaller than the inside boundaries of the shield

Type 2 (A2.5.2)

- Is a flask support assembly with electric heating.
- Consists of an adjustable system onto which the electric heater is mounted.
  - provision for placement of a flask support board above the electric heater
  - Is adjustable from the outside of the shield.

Flask Support Board (A2.6)

- Made of ceramic or other heat-resistant material.
- 3 mm to 6 mm in thickness.
- Shall be classified into A, B, or C category based on the size of the centrally located opening.
- Shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening
- Minimize extraneous heat to the flask other than through the central opening.

Cautionary Note

- Asbestos-containing materials shall not be used in the construction of the flask support board.
- The detailed placement of the flash support board is explained in the Annex A2.7 and A2.8.

Receiving Cylinders (A2.9, A2.9.1, A2.9.2)

- Shall measure and collect 100 mL ± 1.0 mL.
• Shape of the base is stable such that the receiver does not topple when placed empty on a surface inclined at an angle of 13 ° from the horizontal.
• Construction requirements for manual and automated method are described in detail in Annex A2.9.1 and A2.9.2.

Receiving Cylinders (A2.9.3)
• May require placement in cooling bath or thermostated air bath

Residue Cylinders (A2.10)
• Has capacity of 5 mL or 10 mL with 0.1 mL graduations.
  o graduations begin at 0.1 mL
• The top of the cylinder may be flared and the other properties conform to ASTM Specification E1272.

Temperature Measuring Device (6.3.1, 6.3.1.1)
• Shall be mercury-in-glass thermometers conforming to ASTM E1.
  o filled with inert gas
  o has graduated stem with enamel backing
  o shall not be used without a verification of the ice point or checked as prescribed in ASTM E1 and ASTM E77 when exposed to an extended period above an observed temperature of 370 °C

Temperature Measuring Device (6.3.2, 6.3.2.1, 6.3.2.2)
• Temperature measurement systems exhibiting the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer shall also be used.
• Use electronic circuitry or algorithms to simulate the temperature lag of the thermometer.
• Alternatively, place the sensor in a casing,
  o with the tip of the sensor covered so that the assembly has a temperature lag time similar to that of a mercury-in-glass thermometer because of adjusted thermal mass and conductivity

Temperature Sensor Centering Device (6.4.1)
• The temperature sensor shall be mounted through a snug-fitting device for mechanically centering the sensor in the neck of the flask without vapor leakage.
• The use of a plain stopper with a hole drilled through the center is not acceptable for the purpose described in Section 6.4.1.

Automated Equipment (6.5)
- Shall be equipped with a device to automatically shut down power to the unit and to spray an inert gas or vapor in chamber, where distillation flask is mounted in the chambers in the event of fire.

**Barometer (6.6, Cautionary Note)**

- Pressure measuring device measures local station pressure with an accuracy of 1 mm Hg.
- Do not take readings from ordinary aneroid barometers, since these are pre-corrected to give sea level readings.

**Sampling, Storage, and Sample Conditioning (7.1)**

- Determine the Group characteristics that correspond to the sample to be tested from Table 1.

**Sampling (7.2, 7.2.1, 7.2.1.1)**

- Sample in accordance with ASTM Practice D4057/ASTM Practice D4177 and as in Table 2.
- For Group 1.
  - maintain the container to below 10 °C by filling the bottle with the cold liquid sample, and discard the first sample
  - maintain the container to below 10 °C for product at ambient temperature
  - pour the sample into the bottle with minimum agitation
  - close the bottle immediately with a tight-fitting closure

**Cautionary Note**

- Do not completely fill and tightly seal a cold bottle of sample since it may break while warming.

**Sampling (7.2.1.2)**

- For Groups 2, 3, and 4.
  - collect the sample at ambient temperature
  - close the sample bottle immediately with a tight-fitting closure after sampling

**Sample Storage (7.3.1, 7.3.2, 7.3.3, 7.3.4, Note 7)**

- Store the samples as indicated in Sections 7.3.2, 7.3.3, and Table 2, if not testing immediately after collection.
- Store all samples away from direct sunlight or sources of direct heat.
- For Groups 1 and 2.
  - store the sample at a temperature below 10 °C
- For Groups 3 and 4.
store the sample at ambient or lower temperature

**Sample Conditioning (7.4.1, 7.4.1.1)**

- Condition the samples to the temperature shown in Table 2 before opening the sample container.
- For Groups 1 and 2.
  - condition the samples to a temperature of less than 10 °C before opening the sample container, except when the sample is immediately tested and already at the prescribed sample temperature described in Table 3

**Sample Conditioning (7.4.1.2, 7.4.1.3)**

- For Groups 3 and 4.
  - heat the sample to 9-21 °C above its pour point prior to analysis, if the sample is not fluid at ambient temperature
  - shake the sample after melting prior to opening the sample container to ensure homogeneity, if the sample is partially or completely solidified during storage
  - temperature ranges shown in Table 3 for the flask and sample do not apply, if the sample is not fluid at room temperature

**Wet Samples (7.5.1, 7.5.2)**

- Samples of materials with water content are not suitable for testing.
- Obtain another sample that is free from suspended water, if the sample is not dry.
- For Groups 1 and 2, remove the suspended water by,
  - maintaining the sample at 0 °C to 10 °C
  - adding approximately 10 g of anhydrous sodium sulfate per 100 mL of sample
  - shaking the mixture for approximately 2 minutes
  - allow the mixture to settle for approximately 15 minutes
  - use a decanted portion of the sample maintained between 1 °C and 10 °C for the analysis once the sample shows no visible signs of water
  - report that the sample was dried by the addition of a desiccant

**Wet Samples (7.5.3)**

- For Groups 3 and 4, remove the suspended water by shaking the sample with anhydrous sodium sulfate or other suitable drying agent.
  - when a water-free sample is not available
- Separate it from the drying agent by decanting.
- report that the sample has been dried by addition of a desiccant.

**Preparation of Apparatus (8.1, 8.2)**
• Prepare the apparatus by choosing the appropriate distillation flask, temperature measuring device, and flask support board as shown in Table 3.
• Bring the temperature of the receiving cylinder, the flask, and the condenser bath to the indicated temperatures shown in Table 3.
• Make provisions so that the temperature of the condenser bath and the receiving cylinder are maintained at the required temperatures.

_Preparation of Apparatus (8.1, 8.2)_

• Place the receiving cylinder in a bath such that,
  o liquid level is at least as high as the 100 mL mark or
  o the receiving cylinder is surrounded by an air circulation chamber

_Preparation of Apparatus (8.2.1, 8.2.2)_

• For Groups 1, 2, and 3, suitable media for low temperature baths include,
  o chopped ice and water
  o refrigerated brine
  o refrigerated ethylene glycol.
• For Group 4, suitable media for ambient and higher bath temperatures include,
  o cold water
  o hot water
  o heated ethylene glycol

_Preparation of Apparatus (8.3)_

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

_Calibration and Standardization (9)_

• Temperature Measurement System
• Automated system

_Calibration and Standardization (9.1, 9.1.1, 9.1.2)_

• Confirmation of the calibration of the temperature measuring systems shall be made.
  o at intervals of not more than six months, and after the system has been replaced or repaired
• Verify the accuracy and the calibration of the electronic circuitry or computer algorithms.
  o using a standard precision resistance bench
• No algorithms shall be used to correct the temperature for lag and the emergent stem effect when performing this verification.

_Calibration and Standardization (9.1, 9.1.1, 9.1.2)_
Calibration and Standardization (9.1.2.1, 9.1.2.2, Note 10)

- The temperature measurement system shall be considered defective and not used for the test.
  - when the temperature reading is not within the values shown in Table 4 for the respective apparatus when verifying by 9.1.2
- Toluene is used as a verification fluid for calibration.
- Reagent grade toluene and hexadecane (cetane) conforms to specifications of Committee on Analytical Reagents of American Chemical Society.
- Other grades shall be used if the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

Calibration and Standardization (9.1.3, 9.1.4, 9.1.5)

- A procedure to determine the magnitude of the temperature lag is described in Annex A3.
- A procedure for the emergent stem effect is described in Appendix X4.
- Use hexadecane to verify the calibration of the temperature measurement system at elevated temperatures.
- Compare the 50 % recovered temperature to that shown in Table 4 for the respective apparatus under Group 4 distillation conditions.

Automated Method (9.2)

- Level Follower
- Barometric Pressure

Level Follower (9.2.1)

- For a level follower/recording mechanism of an automated distillation apparatus, a resolution of 0.1 volume % or better with a maximum error of 0.3 volume % between 5 and 100 volume % is required.
- Verify calibration of the assembly in accordance with manufacturer’s instructions at intervals of not more than three months and after the system has been replaced or repaired.

Barometric Pressure (9.2.1)

- Verify barometric reading of the instrument against a barometer, as described in Section 6.6, at intervals of not more than six months and after the system replacement or repair.