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

Procedure

Note

- This presentation covers those aspects of ASTM D86 which are specific to test procedure.
- For specific details regarding the equipment refer to the other presentation in this training module.

Procedure

Procedure (10.1, 10.2)

- Record the prevailing barometric pressure.
- For Groups 1 and 2,
  o ensure that the sample is conditioned in accordance with Table 2
  o fit a low range thermometer provided with a snug-fitting cork or stopper of silicone rubber, or equivalent polymeric material, tightly into the neck of the sample container
  o bring the temperature of the sample to the temperature indicated in Table 3

Procedure (10.3)

- For Groups 1, 2, 3, and 4,
  o check that the temperature of the sample is as shown in Table 3
  o pour the specimen precisely to the 100 mL mark of the receiving cylinder
  o transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that no liquid flows into the vapor tube

Procedure (10.3.1)

- For Groups 3 and 4,
  o heat the sample to a temperature between 9 °C and 21 °C above its pour point prior to analysis, if the sample is not fluid at ambient temperature
  o shake the sample after melting, and prior to sampling, to ensure homogeneity of the sample, if the sample is partially or completely solidified in the intervening period

Procedure (10.3.1.1)

- Disregard the temperature range shown in Table 3 for the receiving cylinder and sample.
o if the sample is not fluid at ambient temperature

- Heat the receiving cylinder to approximately the same temperature as the sample prior to analysis.
- Pour the heated specimen precisely to the 100 mL mark of the receiving cylinder.
- Repeat the transferring step as described in Section 10.3.

**Procedure (10.4)**

- Add a few boiling chips to the specimen, if the sample is expected to demonstrate irregular boiling behavior, such as bumping.
  o addition of a few boiling chips is acceptable for any distillation

**Procedure (10.5)**

- Fit the temperature sensor through a snug-fitting device,
  o as described in 6.4, to mechanically center the sensor in
  o the neck of the flask.
- In the case of a thermometer, place the bulb in the neck and level the lower end of the capillary with the highest point on the bottom of the inner wall of the vapor tube
- In the case of a thermocouple or resistance thermometer, follow the manufacturer’s instructions for placement

**Procedure (10.6)**

- Fit the flask vapor tube with a snug-fitting cork or rubber stopper of silicone, or equivalent polymeric material tightly into the condenser tube.
- Adjust the flask in a vertical position so that the vapor tube extends into the condenser tube for a distance from 25 mm to 50 mm.
- Raise and adjust the flask support board to fit it snugly against the bottom of the flask.

**Procedure (10.7)**

- Place the used receiving cylinder, without drying the cylinder, under the lower end of the condenser in its temperature controlled bath.

**Initial Boiling Point (10.8)**

- Manual Method
- Automated Method

**Manual Method (10.8.1)**

- Cover the receiving cylinder with a piece of blotting paper, or similar material, to fit the condenser tube snugly.
  o to reduce evaporation loss of the distillate
• Start the distillation with the tip of the deflector just touching the wall of the receiving cylinder.
  o if a receiver deflector is used
• Keep the drip tip of the condenser away from the wall of the receiving cylinder.
  o if a receiver deflector is not used

**Manual Method (10.8.1)**

• Note the start time.
• Observe and record the IBP temperature to the nearest 0.5 °C. Keep the drip tip of the condenser away from the wall of the receiving cylinder.
• Immediately move the receiving cylinder so that the tip of the condenser touches the inner wall
  o if a receiver deflector is not used

**Automated Method (10.8.2)**

• Use the device provided by the instrument manufacturer to reduce evaporation loss of the distillate.
• Apply heat to the distillation flask and contents with the tip of the receiver deflector just touching the wall of the receiving cylinder.
• Note the start time.
• Record the IBP to the nearest 0.1 °C.

**Procedure (10.9, 10.10, 10.11)**

• Regulate the heating process,
  o so that the time interval between the first application of heat and the IBP is as specified in Table 5
  o and the time from IBP to 5 % recovered is as indicated in Table 5
• Continue regulation of heat so that the uniform average rate of condensation from 5 % recovered to 5 mL residue in the flask is 4 mL to 5 mL per min.

**Cautionary Note**

• The distillation rate shall be kept as constant as possible.
  o The distillation rate has an effect on the measured vapor temperature

**Procedure (10.12, 10.13, 10.14)**

• Repeat any distillation process when the sample does not meet the requirements as described in Sections 10.9, 10.10, and 10.11.
• Discontinue the heating procedure and proceed as directed in Section 10.17 when a decomposition point, as described in Section 3.1.3, is observed.
• Observe and record data necessary for the calculation and reporting, in the interval between the IBP and the end of the distillation.
• Report the results of the test as required by the specification involved, or as previously established for the sample under test.
• Include temperature readings for observed data at prescribed percentages recovered, or percentages recovered at prescribed temperature readings.

Procedure (10.14.1, 10.14.2)

• For Manual Method,
  o Record all volumes in the graduated cylinder to the nearest 0.5 mL.
  o Record all temperature readings to the nearest 0.5 °C.
• For Automated Method,
  o Record all volumes in the receiving cylinder to the nearest 0.1 mL.
  o Record all temperature readings to the nearest 0.1 °C.

Procedure (10.14.3, 10.14.3.1)

• For Groups 1, 2, 3, 4,
  o record the IBP and the EP (FBP) or the dry point, or both, and temperature readings at 5 %, 15 %, 85 %, and 95 % recovered, and at each 10 % multiple of volume recovered from 10 to 90, inclusive.
• For Group 4, when pertinent thermometer readings are obscured
  o perform a second distillation in accordance with Group 3
  o report the reading from the low range thermometer readings in place of the obscured high range thermometer readings
  o indicate these on the test report.

Procedure (10.14.4)

• Record temperature readings at every 1 % recovered when,
  o the temperature reading is reported at a prescribed percent evaporated or recovered for a sample that has rapidly changing slope of the distillation curve in the region of the prescribed percent evaporated or recovered reading.
• The slope is considered rapidly changing if,
  o the change in slope (C) of the data points described in Section 10.14.2 in that particular area is greater than 0.6 as calculated using the formulae for change of slope.

Procedure (10.14.4)

• Change of Slope, \( C = \frac{(C_2 - C_1)}{(V_2 - V_1)} - \frac{(C_3 - C_2)}{(V_3 - V_2)} \)
where,

\[ C_1 = \text{temperature at the volume \% recorded one reading prior to the volume \% in question, } ^\circ\text{C} \]
\[ C_2 = \text{temperature at the volume \% recorded in question, } ^\circ\text{C} \]
\[ C_3 = \text{temperature at the volume \% recorded following the volume \% in question, } ^\circ\text{C} \]
\[ V_1 = \text{volume \% recorded one reading prior to the volume \% in question} \]
\[ V_2 = \text{volume \% recorded at the volume \% in question, and} \]
\[ V_3 = \text{volume \% recorded following the volume \% in question} \]

**Procedure (10.15)**

- Make a final adjustment of the heat, when the residual liquid in the flask is approximately 5 mL.
- The time from the 5 mL of liquid residue in the flask to the EP (FBP) shall be within the limits as prescribed in Table 5.
- Repeat the test with appropriate modification of the final heat adjustment, if this condition is not satisfied.

**Procedure (10.15.1, 10.16)**

- Rerun the test, if the actual front end loss differs more than 2 mL from the estimated value.
- Observe and record the EP (FBP) or the dry point
- Discontinue the heating process.
- Allow the distillate to drain into the receiving cylinder after heating is discontinued.

**Procedure (10.17.1, 10.17.2)**

- For Manual Method,
  - observe and note the volume of condensate to the nearest 0.5 mL at 2 minute.
  - intervals until two successive observations agree while the condenser tube continues to drain into the graduated cylinder
  - measure the volume in the receiving cylinder accurately
  - record it to the nearest 0.5 mL
- For Automated Method,
  - monitor continually the recovered volume of the receiving cylinder until the volume changes by no more than 0.1 mL in 2 minutes
- Record the volume in the receiving cylinder accurately to the nearest 0.1 mL

**Procedure (10.18)**

- Record the volume in the receiving cylinder as percent recovery.
- Deduct the percent recovered from 100 %, if the distillation is previously discontinued under the conditions of a decomposition point.
- Report this difference as the sum of percent residue and percent loss.

**Procedure (10.19)**

- Disconnect the distillation flask from the condenser
- Pour the remaining contents of the flask into the 5 mL graduated cylinder until no increase in volume is observed
- Record the volume in the graduated cylinder as percent residue.

**Procedure (10.19.1, 10.19.1.1)**

- Prefill the cylinder with 1 mL of a heavy oil to allow better estimate of the volume of the material recovered.
  - if the 5 mL graduated cylinder does not have graduations below 1 mL and
  - the volume of liquid is less than 1 mL
- Check whether adequate heat is applied towards the end of the distillation and whether conditions during the test conformed to those specified in Table 5, if
  - a residue greater than expected is obtained and
  - the distillation is not purposely terminated before the EP

**Procedure (10.19.2, 10.20, 10.21)**

- Record the volume in the 5 mL graduated cylinder to the nearest 0.1 mL as percent residue for Groups 1, 2, 3 and 4.
- Modify the procedure to conform to the instructions described in Annex A4, if
  - the intent of the distillation is to determine the percent evaporated or percent recovered at a predetermined corrected temperature reading
- Examine the condenser tube and the side arm of the flask for waxy or solid deposits.
  - Repeat the test after making adjustments described in Footnote A of Table 5.

**Calculation (11.1, 11.2, 11.3)**

- Calculate the percent total recovery with the formula,
- Percent total recovery = percent recovery + percent residue
- Deduct the percent total recovery from 100 to obtain the percent loss.
- Do not correct the barometric pressure for meniscus depression.
- Do not adjust the pressure to sea level.
- Correct temperature readings to 101.3 Kpa pressure.

**Calculation (11.3)**

- Obtain the correction to be applied to each temperature reading by means of the Sydney Young equation using the following formulae, or by the use of Table 6.

For Celsius temperatures,

\[ C_c = 0.0009(101.3 - P_k)(273 + t_c) \]
\[ C_c = 0.00012(760 - P)(273 + t_c) \]

**Calculation (11.3)**

**Calculation (11.3, 11.4)**

- Use the corrected temperature readings in all further calculations and reporting after applying the corrections.
- Rounding each result to the nearest 0.5 °C or 0.1 °C, as appropriate to the apparatus being used.
- Correct the actual loss to 101.3 kPa pressure when temperature readings are corrected to 101.3 kPa pressure.

**Calculation (11.3, 11.4)**

Calculate corrected loss \( L_c \) using the formula,

\[ L_c = 0.5 + \frac{L - 0.5}{1 + \frac{101.3 - P_k}{8.00}} \]
\[ L_c = 0.5 + \frac{L - 0.5}{1 + \frac{760 - P}{60.00}} \]

where,

- \( L \) = observed loss
- \( L_c \) = corrected loss
- \( P_k \) = pressure, kPa
- \( P \) = pressure, mm Hg

**Calculation (11.4.1)**

Calculate the corresponding corrected percent recovery in accordance with the following equation:

\[ R_c = R + (L - L_c) \]

where,
\[ \begin{align*} 
L &= \text{percent loss or observed loss} \\
L_c &= \text{corrected loss} \\
R &= \text{percent recovery} \\
R_c &= \text{corrected percent recovery} 
\end{align*} \]

**Calculation Percent Evaporated at a Prescribed Temperature Reading (11.5, 11.6)**

- Add the percent loss to each of the observed percent recovered at the prescribed temperature readings to obtain the percent evaporated at a prescribed temperature reading.
- Report these results as the respective percent evaporated.
  \[ \text{Pe} = \text{Pr} + L \]
  where,
  \[ \begin{align*} 
  L &= \text{observed loss}, \\
  \text{Pe} &= \text{percent evaporated}, \text{ and} \\
  \text{Pr} &= \text{percent recovered} 
\end{align*} \]
- Use either arithmetical procedure or graphical procedure to obtain temperature readings at prescribed percent evaporated.
  - when no recorded temperature data is available within 0.1 volume % of the prescribed percent evaporated
- Indicate the procedure used on the report.

**Arithmetical Procedure (11.6.1)**

- Deduct the observed loss from each prescribed percent evaporated to obtain the corresponding percent recovered.
- Calculate each required temperature reading as follows,
  \[ T = TL + (TH - TL) (PR - PRL)/(PRH - PRL) \]
  where,
  \[ \begin{align*} 
  PR &= \text{percent recovered corresponding to the prescribed percent evaporated} \\
  PRH &= \text{percent recovered adjacent to, and higher than PR} \\
  PRL &= \text{percent recovered adjacent to, and lower than PR} \\
  T &= \text{temperature reading at the prescribed percent evaporated}, 
\end{align*} \]
TH = temperature reading recorded at RH
TL = temperature reading recorded at RL

**Graphical Procedure (11.6.2)**

- Use graph paper with uniform subdivisions.
- Plot each temperature reading corrected for barometric pressure against its corresponding percent recovered.
- Plot the IBP at 0 % recovered.
- Draw a smooth curve connecting the points.
- Deduct the distillation loss to obtain the corresponding percent recovered and take from the graph the temperature reading that this percent recovered indicates for each prescribed percent evaporated.
- Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

**Calculation Percent Evaporated at a Prescribed Temperature Reading Automated Procedure (11.6.3)**

- Collect temperature-volume data at 0.1 volume % intervals or less in most automated instruments.
- Store the data in memory.
- Report a temperature reading at a prescribed percent evaporated, neither of the procedures described in Sections 11.6.1 and 11.6.2 to be used.
- Obtain the desired temperature directly from the database as the temperature closest to and within 0.1 volume % of the prescribed percent evaporated.

**Report (12.2, 12.3, 12.3.1, 12.3.2)**

- Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).
- For Manual Method,
  - report volumetric readings to the nearest 0.5 %
  - all temperature readings to the nearest 0.5 °C
- For Automated Method,
  - report volumetric readings to the nearest 0.1 %
  - all temperature readings to the nearest 0.1 °C

**Report (12.4.1, 12.5, 12.6)**

- Report if the temperature readings are not corrected for barometric pressure.
- Report the percent residue and percent loss as “observed” in accordance with Sections 10.19 and 11.1, respectively when,
o when the temperature readings are not corrected to 101.3 kPa pressure

Report (12.7,12.8)

- Take the result based on relationships between temperature readings and percent evaporated when,
  - the sample is a gasoline
  - any other product classified under Group 1, in which the percent loss is greater than 2.0
- Otherwise, the report can be based on relationships between temperature readings and percent evaporated or percent recovered.
- In the manual method, if results are given in percent evaporated versus temperature readings, report if the arithmetical or the graphical procedure was used.
- Report if a drying agent was used.