

B. GENERAL TESTS

Boiling Point and Amount of Distillate

Unless otherwise specified, the boiling point and amount of distillate are determined by Method 1 or Method 2 below as appropriate.

Unless otherwise specified, the minimum boiling point is expressed as the temperature at which the first 5 drops of distillate are obtained and the maximum boiling point is expressed as the temperature, just before the liquid in the distillation flask is almost gone and a sufficient amount of vapor is no longer obtained.

The amount of distillate is the measured volume of the distillate that is evaporated within the specified temperature range.

Hereinafter in the Monographs, such a specification as "55.5 - 57.0 (Method 1)" indicates that when determined as directed in Method 1 under Boiling Point and Amount of Distillate, the boiling point of the substance is 55.5 - 57.0 . Also, such a specification as "the amount of distillate at 64 - 70 is not less than 95% vol. (Method 2)" indicates that when determined as directed in Method 2 under Boiling Point and Amount of Distillate, the amount of distillate of the substance at 64 - 70 is not less than 95% vol.

Method 1 This method is used to determine the boiling point and amount of distillate of a liquid when the specified temperature range is less than 5 .

Apparatus Use the apparatus illustrated on the next page.

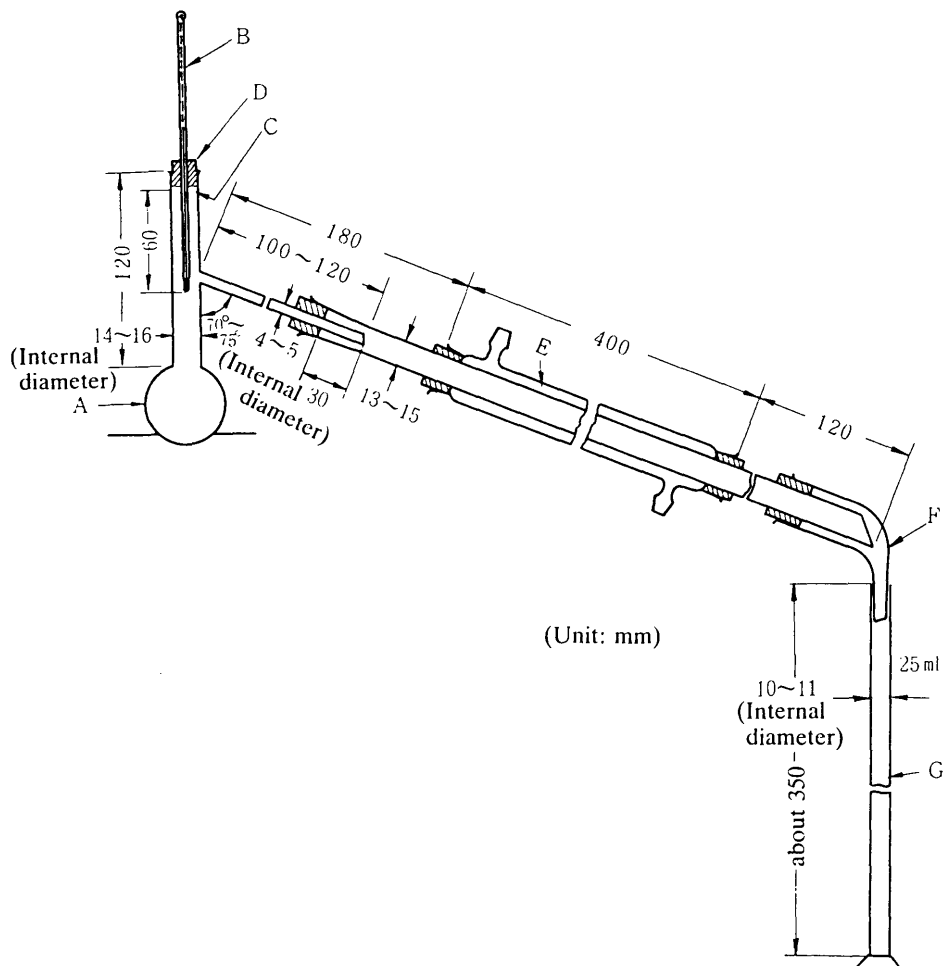
Dry thoroughly all glass instruments before use. Insert thermometer with immersion line B into the distillation flask so that immersion line C is on a level with the lower end of cork stopper D and the upper end of its mercury bulb is located in the center of the delivery tube. Connect condenser E with distillation flask A, adapter F with E, then insert the open end of F into the mouth of G so that air passes through slightly.

Place boiling tips or a capillary tube into A, use a hood with a height sufficient to shield A, and heat A with a suitable heat source. When direct flame is applied as the heat source, place A on a hole of a ceramics board (a wire gauge of 150 mm square covered with ceramics of 6-mm thick with a round hole of 30 mm in diameter in its center), and then heat.

Procedure Measure 25 ml of the sample, whose temperature is previously measured, using G, and transfer into A. Use G as the receiver without washing. When the apparatus is set up, pass water through E, heat A, and start distillation after about 10 minutes. Unless otherwise specified, distill the liquid sample by the application of heat, at a rate of 4 to 5 ml per minute in the case of liquids whose boiling temperature to be determined is less than 200 , and at a rate of 3 to 4 ml per minute in the case of liquids whose boiling temperature is not less than 200 . Lower

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the temperature of distillate to the temperature at which the liquid was originally measured, and measure the volume of distillate.



- A: Hard glass distillation flask (Capacity: 50 - 60 ml)
- B: Thermometer with an immersion line (rod)
- C: Immersion line
- D: Stopper
- E: Condenser
- F: Adapter
- G: Volumetric cylinder (25 ml, graduated in 0.1-ml divisions)

Liquids that begin to distill at 80 °C or below are cooled to 10 °C to 15 °C before measuring the volume, and receiving cylinder G is kept immersed in ice up to a point 25 mm from the top during the distillation.

Correct the measured temperature for any variation in the barometric pressure from the normal (101kPa) by allowing 0.1 °C for each 0.36kPa of variation, adding if

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the pressure is lower than 101kPa, or subtracting if higher than 101kPa.

Method 2 This method is used to determine the boiling point and amount of distillate of a liquid when the specified range of temperature is not less than 5 .

Apparatus The same apparatus as described in Method 1 is used. Use 200-ml distillation flask A, the neck of which is 18 to 24 mm in internal diameter, and which has a delivery tube having the internal diameter of 5 to 6 mm. The ceramics board used for direct-flame heating should have in its center a round hole of 50 mm in diameter.

For volumetric cylinder G used as the receiver, use one that has a capacity of 100 ml and a scale graduated in 1-ml divisions.

Procedure Measure 100 ml of the sample, whose temperature is previously noted, using G graduated in 1-ml divisions, and carry out the distillation in the same manner as Method 1.