

1.2.4 Boiling range

The boiling range (distillation range) is the corrected range of temperature, within which the whole or a specified portion of a liquid distils, under normal atmospheric pressure, when determined by the method described below.

Apparatus

A suitable apparatus for the determination consists of a distillation flask, a condenser, a receiver, a heat source with heat shields, and a thermometer.

The distillation flask of 50-60 mL capacity is made of heat-resistant glass. Flasks with the following dimensions are suitable: neck 10-12 cm long and 14-16 mm in internal diameter with a side-arm, 10-12 cm long and about 5 mm in internal diameter, attached at about the midway point of the neck and forming an angle of 70-75° with the lower portion of the neck.

The condenser is either a straight glass condenser, made of heat-resistant glass, 55-60 cm in length with a water jacket about 40 cm in length, or a condenser of another design having equivalent condensing capacity. The lower end of the condenser may be bent to provide a delivery tube, or a bent adapter may be attached to it to serve as a delivery tube.

The receiver consists of a 25-50-mL measuring cylinder graduated in subdivisions of 0.5 mL.

The heat source consists of a small gas burner, preferably of a Bunsen type, or an electric heater or mantle capable of adjustment comparable to that possible with the gas burner. If a gas burner is used, a suitable shield is placed around the flask near its bottom. The shield is made of heat-resistant material, in the form of a square with sides of 14-16 cm and with a perforation in the centre. The diameter of the latter should be such that when the flask is set into it, the portion of the flask below the upper surface of the shield has a capacity of 3-4 mL.

The thermometer should preferably be calibrated for partial immersion of 100 mm as described under A for the determination of melting temperature and melting range of pulverizable substances; otherwise a total-immersion thermometer may be used with appropriate emergent-stem correction. When the thermometer is placed in position, the stem should be located in the centre of the neck of the flask, and the top of the bulb should be just below the bottom of the outlet to the side-arm.

Recommended procedure

Place in the flask 25 mL of the liquid to be tested, taking care not to allow any of the liquid to enter the side-arm, and add 0.3-0.5 g of glass-beads or other suitable substance. Shield the burner and the flask from external air currents and apply heat so that the vapour rises only slowly into the neck of the flask and between 5-10 minutes elapse before the first drop of distillate falls from the condenser. Continue the distillation at the rate of 2-3 mL per minute, collecting the distillate in the receiver. Read the temperature when the first drop of distillate falls from the condenser, and again when the last quantity of liquid evaporates from the bottom of the flask or when the specified percentage has distilled over.

The boiling ranges (distillation ranges) indicated in the monographs are applied at a barometric pressure of 101.3 kPa (760 mmHg). If the determination is made at some other barometric pressure, correct the observed temperature readings for any difference in the barometric pressure by allowing 0.1 °C for each 0.36 kPa (2.7 mmHg) of the difference, adding if the pressure is lower, or subtracting if the pressure is higher.