



## B. GENERAL TESTS

- E: Thermometer fastening spring
- F: Vent for adjustment of the bath fluid volume
- G: Coiling spring
- H: Capillary tube (use a hard glass capillary tube 0.8 to 1.2 mm in internal diameter, 120-mm long, and walls 0.2 to 0.3 mm thick, whose one end is closed.
- J: Spring for fastening Teflon stopper  
Immersion line (Unit: mm)

**Procedure** Reduce a sample into a fine powder and, unless otherwise specified, dry in a desiccator for about 24 hours. When the stipulation “dried substance” is given in the Monographs, dry the sample under the conditions specified in Loss on Drying in the individual monograph, and fill capillary tube H with the sample as tightly as practical to form a layer of 2.5 to 3.5 mm in height. When the stipulation “sealed tube” is given in the Monographs, close one open end of the tube. When the stipulation “vacuum sealed tube” is given, close one open end of the tube by heating gently under reduced pressure not exceeding 0.67kPa.

Heat bath fluid B slowly until the temperature rises to about 10 °C below the expected melting point, adjust the immersion line of thermometer D at the same level as the meniscus of the bath fluid, and insert capillary tube H into coil spring G so that the part of H containing the sample is on a level with the middle of the mercury bulb of thermometer D. Then, continue heating so that the temperature rises at a rate of about 3 °C per minute until the temperature rises to about 5 °C below the expected melting point, and continue heating at the rate of increase of 1 °C per minute.

The temperature at which slight wetting or disintegration is observed in the contact part of the inside wall of H and the sample is designated as the beginning of melting, and the temperature at which the sample melts completely and becomes transparent is designated as the end of melting. The temperature of the end of the melting is designated as the melting point.

### (2) Procedure for Class 2 Substances

**Procedure** Melt the sample at a temperature as low as practical, and suck it into the capillary tube (the same one as used in (1) and which is open at both ends) to about 10 mm in height. Allow to stand at about 10 °C for 24 hours or cool for at least 2 hours with ice. Attach the tube to the thermometer by a rubber band at the position the part of the tube containing the sample is on a level with the middle part of the mercury bulb. Set it in a beaker with water to such a position that the upper edge of the sample is about 10 mm below the water surface. Heat the water with constant stirring until the temperature rises to about 5 °C below the expected melting point, and continue heating at the rate of increase of 1 °C per 2 minutes. The temperature at which the sample is observed to rise in capillary tube H is designated as the melting point.