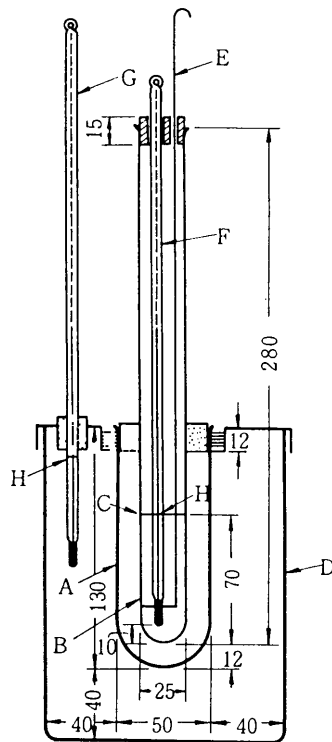


B. GENERAL TESTS

Congealing Point

Congealing Point is measured as directed below.

Apparatus Use the apparatus illustrated in Fig. 1.



(Unit: mm)

- A: Glass cylinder (Silicone oil is applied to both sides of the wall to prevent misting).
- B: Sample container (A hard-glass test tube to both sides of which silicone oil is also applied to prevent misting, excluding parts in contact with the sample. Insert it into cylinder A, and fix with a cork stopper).
- C: Marked line.
- D: Glass or plastic cooling bath.
- E: Glass or stainless steel stirring rod (A 3-mm diameter rod with the lower end bent into a loop about 18 mm in diameter).
- F: Rod thermometer with an immersion line.
- G: Auxiliary thermometer
- H: Immersion line

Fig. 1

B. GENERAL TESTS

Procedure Fill glass or plastic cooling bath D almost to the top with water at a temperature about 5° lower than the expected congealing point. If the sample is a liquid at ordinary temperature, fill bath D with water at a temperature 10 - 15° lower than the expected congealing point. Transfer the sample into sample container B up to marked line C. If the sample is a solid, melt carefully the sample by warming to a temperature lower than 20° above the expected congealing point, and transfer into B. Insert B into glass cylinder A, and adjust immersion line H of thermometer F so that it is on a level with the meniscus of the sample. After cooling the sample to a temperature 5° higher than the expected congealing point, move vertically stirring rod E at a rate of 60 to 80 strokes per minute, and observe the temperature readings at 30-second intervals. The temperature falls gradually. Stop stirring when an appreciable amount of crystals are formed and the temperature becomes constant, or begins to rise.

Ordinarily, the temperature becomes constant for a short time after rising. Read the maximum temperature (reading of F) that is kept constant (Fig. 2). If no rise of temperature occurs, read the temperature remained constant for a while (Fig. 3). The congealing point is the average of four or more consecutive readings, between which the temperature difference is within 0.2°.

If a lot of impurities are included in the sample, the congealing point curve is not as shown in Fig. 2, but as shown in Fig. 3, 4, or 5. In the case of Figs. 4 and 5, extend each of the curves of the solid and liquid phases on the graph, and take their intersection as the congealing point. In the case of Fig. 3, proceed as in Fig. 2.

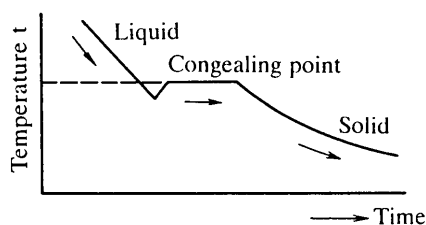


Fig. 2

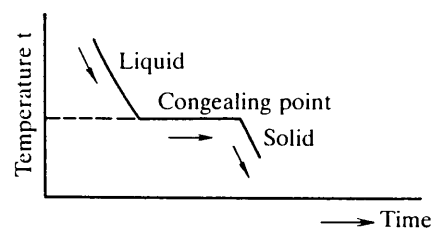


Fig. 3

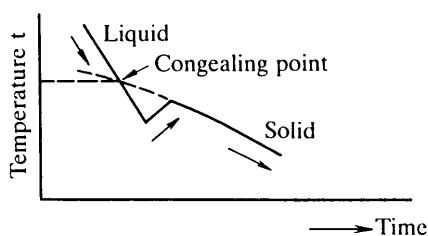


Fig. 4

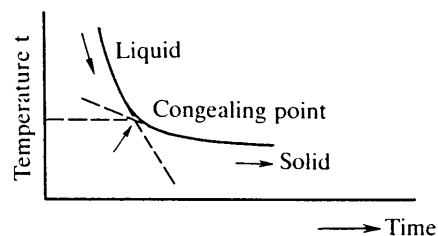


Fig. 5

Note: If it is anticipated to be the state of super cooling, rub the inner wall of bath B or put a small fragment of the solid sample into bath B for promoting the congealment when the temperature approaches the expected congealing point.