



## MELTING POINT APPARATUS

### Preparation of samples for melting point determination

#### • Introduction

The melting point of a substance is the point at which the solid phase converts to a liquid phase at 1 atmosphere of pressure. The melting point is usually recorded as a range, with the lower temperature the point at which liquid is first visible in the sample and the upper temperature when the last solid has completely melted. Pure substances usually melt in a range of two degrees or less. If a sample is impure, its melting point will be lower or wider than the expected published value.

#### • Equipment and reagents

Sample  
Watch glass  
Glass rod  
Melting point capillary tubes

#### • Method

1. For accurate melting point determinations, ensure the sample is dry. Dry the sample overnight in a vacuum desiccator with desiccant if necessary.
2. Place a small quantity of the dry sample on a watch glass and grind to a fine powder with a glass rod. Coarse, crystalline samples will give uneven heat transfer through the sample.
3. The glass capillary will need to be sealed at one end; if the capillary is open at both ends, seal one of the ends using a Bunsen burner.
4. Gather the powdered sample into a small pile. Press the open end of the capillary into the sample to a depth of around 2mm.
5. Invert the capillary and tap the tube to pack the sample down in the sealed end. Repeat as necessary; the final height of the sample should be between 2-4mm.
6. If preparing multiple samples, prepare them in the same way with the same height of sample in the tube. For accurate melting point determinations it is recommended that three samples of the same material are prepared in the same way and analyzed at the same time. The average of the results from the three capillaries can then be reported as the melting point of the sample.

#### • Tips

- Ensure there is no sample on the outside of the tube before inserting it into the melting point apparatus.
- A sample should be used only once for melting point determination as some substances chemically decompose on heating.
- Do not heat the sample too fast; generally, 1-2°C/minute is recommended.
- If the melting temperature is unknown, first perform a rapid melt with a ramp of up to 20°C/minute. Once the approximate melting point is known, a more appropriate plateau can be set and a slower ramp rate used.