

# DETERMINATION OF MELTING POINTS

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# Background

- The melting point of a substance is the temperature at which the material changes from a solid to a liquid state. Pure crystalline substances have a clear, sharply defined melting point. During the melting process, all of the energy added to a substance is consumed as heat of fusion, and the temperature remains constant.
- A pure substance melts at a precisely defined temperature, characteristic of every crystalline substance and dependent only on pressure (though the pressure dependency is generally considered insignificant).
- Determining the MP is a simple and fast method used in many diverse areas of chemistry to obtain a first impression of the purity of a substance. This is because even small quantities of impurities change the melting point, or at least clearly enlarge its melting range. Melting point determinations are more than just a classroom exercise in the organic chemistry lab. The test is still an important technique for gauging purity of organic and pharmaceutical compounds.

- The determination of melting points is one of the oldest identification and test methods for organic substances. The melting point is easy to measure, tabulate and classify. Extensive collections of tables give the exact values of many pure, inorganic and organic compounds. The MP determination is a fast and cost-effective technique which remains a strong link to the vast pre-instrumental chemistry literature.

# Sample preparation

Any substance being loaded into a melting point capillary must be:

1. Fully dry
2. Homogeneous
3. In powdered form

Moist samples must be dried first—48 hours over  $P_2O_5$ , in a dessicator, usually gets the job done. The primary requirement for good melting point determination is that the sample be in a fine powder form. This makes the heat transfer into the sample more efficient and reproducible, and also enhances the overall reflectivity of the sample for easier automated detection of the melt. Coarse crystalline and non-homogeneous samples must be crushed into a fine powder in a mortar. An agate, glass or alumina mortar and pestle are recommended.

Solid samples, mortar and pestle, and capillary tubes—the basic ingredients for sample preparation



# Procedure

- 1- Take a capillary tube and sealed an open end of capillary tube by inserting the tip into a Bunsen flame near the base of the flame
- 2- Place a small amount of the compound (Benzoic acid) in a clean surface. Push the open end of the capillary tube into the compound
- 3- Move the powder to the closed end of the capillary tube by tapping it on the tables . Repeat until the compound occupies 1-2 mm of the capillary tube end
- 4-place the capillary tube inside the melting point device and observe the changes that occur to the substance as the temperature rises
- 5-Note the temperature at which the compound melts and Record the temperature at which the solid in the capillary tube melts

# Melting point device

