THE DETERMINATION OF MELTING POINTS

Pure crystalline substances have a fixed and sharply defined temperature at which they change from solid to liquid. Since the melting process requires thermal energy input to disrupt the crystal lattice, the temperature remains constant while melting takes place, provided that the two phases are in equilibrium. In a laboratory melting point determination, this condition is not usually achieved and the melting of the sample is observed to occur over a small temperature range, typically 1 – 2 degrees Celsius. The situation changes when the sample is not completely pure, and the presence of impurities in the sample will cause melting to occur at a lower temperature and over a broader range. This depression of the melting temperature increases as the amount of impurity increases, making the observation of a sample's melting range a useful indicator of its purity. (This does not apply to some substances which decompose before reaching their melting point). The melting point depression phenomenon can be applied to testing two samples to confirm whether they are the same substance. If a mixture of approximately equal parts of the two samples melts at the same temperature as the two individual samples, then it is almost certain that they are identical. (There are a few rare exceptions, but these can be eliminated by testing further mixtures of different proportions.)

In practice, the melting point test is carried out on a small amount of finely powdered substance contained in a length of 1.5 mm diameter capillary tubing which has been sealed at one end by melting in a gas flame. The depth of the sample in the tube should be 2 – 3 mm after packing the sample in the sealed end by tapping the tube on the bench. It is essential that the sample is completely dry and free from residual solvents. The tube containing the sample is then slowly heated in a suitable apparatus while closely observing the sample for changes. As the temperature nears the melting point, the tiniest crystals adhering to the wall of the capillary tube will be seen to melt first, followed shortly afterwards by the bulk of the sample beginning to shrink, collapse and liquefy. This temperature is recorded as the **INITIAL MELTING POINT**. Soon a meniscus forms at the top of the sample, while there is still some solid in the lower portion. The solid portion rapidly decreases until the moment when the sample becomes entirely liquid. This is recorded as the **FINAL MELTING POINT**.

It is normal to carry out a preliminary test at a fairly rapid heatup rate, and then repeat the test approaching the melting point more slowly for a more accurate result. Once a sample has been melted, it can not be re-used. A fresh sample must be prepared for a duplicate test.