## *Using Melting Point to Determine Purity of Crystalline Solids*

When an organic solid is heated, the *heat energy* that's added to the substance is translated into *kinetic energy* – the movement of the molecules. The more mobile molecules are able to partially overcome the intermolecular attractive forces which keep them adhered rigidly in place in the highly-ordered structure of the crystalline "lattice." The individual molecules can move more freely in the liquid state, and the interactions between them are transient in nature.

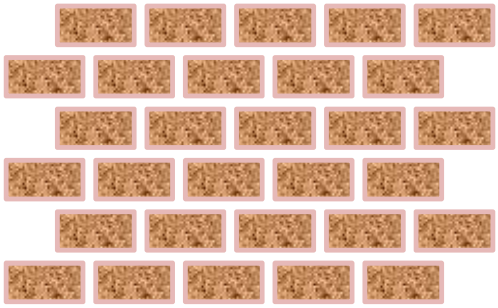
The ***melting point*** of a substance is the *temperature range* over which *the first crystal* of a solid *just starts to melt* and the *last crystal completes its melting*.

A melting point range is very ***narrow*** for ***pure*** solids (usually just 1 – 2 $C^{o}$), and it is an *intensive* physical property – *characteristic* of the particular compound. Thus a melting point can be used to tentatively *identify pure compounds* in their solid state.

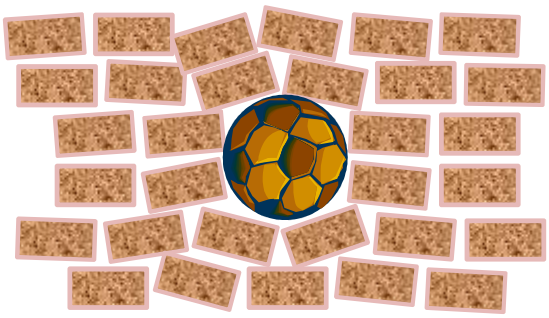
The presence of even a small amount of *impurity* will ***lower*** a compound's melting point by a few degrees and ***broaden*** the melting point temperature range. Because the impurity causes defects in the crystalline lattice, it is easier to overcome the intermolecular interactions between the molecules.

To better understand this concept, you can imagine the crystalline structure of an organic solid as being like a brick wall. Each brick in the wall represents an organic molecule, and the mortar that holds the bricks together is the intermolecular attractive forces.

In a very *pure* organic crystal, all of the molecules are the same, so they pack together in a perfect, very orderly array. In this array, the attractive forces between the molecules are maximized. The brick wall (on the left) symbolizes this ideal crystalline structure – all of the bricks (the molecules) are identical, and they adhere tightly together in an orderly, three-dimensional array.



***Pure "Crystal" Structure***



***Impure "Crystal" with "Defect"***

But with a *mixture* of two different organic molecules, the crystals would be impure. These *different* molecules would *not* fit together properly to make an orderly array.

Thinking in terms of our brick wall analogy, you can imagine trying to mortar a soccer ball into place within the brick wall. There's nothing right about that soccer ball (that different molecule): it's the wrong size, the wrong shape, and it doesn't adhere well with the mortar (it has the wrong intermolecular attractive forces). It creates a defect in the structure of the brick wall (a defect in the crystal), so the structure is weak, and it is easily overcome by an input of energy.

*Less heat* is needed to melt this *mixture* than is required to melt the *pure structure*. Less heat corresponds to a *lower* temperature; thus, *an impure solid melts at a lower temperature* than the same solid with no impurities present. The impure solid also melts over a *broader temperature range*, due to regions within the crystal that contain different amounts of the impurity, and thus different numbers of defects in the crystalline brick wall.

## *Melting Point Determination*

### *Sample Preparation*

1. As demonstrated in the LabCam video, tap the *open* end of a glass capillary tube into a finely-powdered solid sample to force some crystals inside.
2. *Invert* the tube, and tap it on the lab bench so the crystals fall to the bottom (the closed end) of the tube. The column of crystals should be only 1 – 2 mm in height.

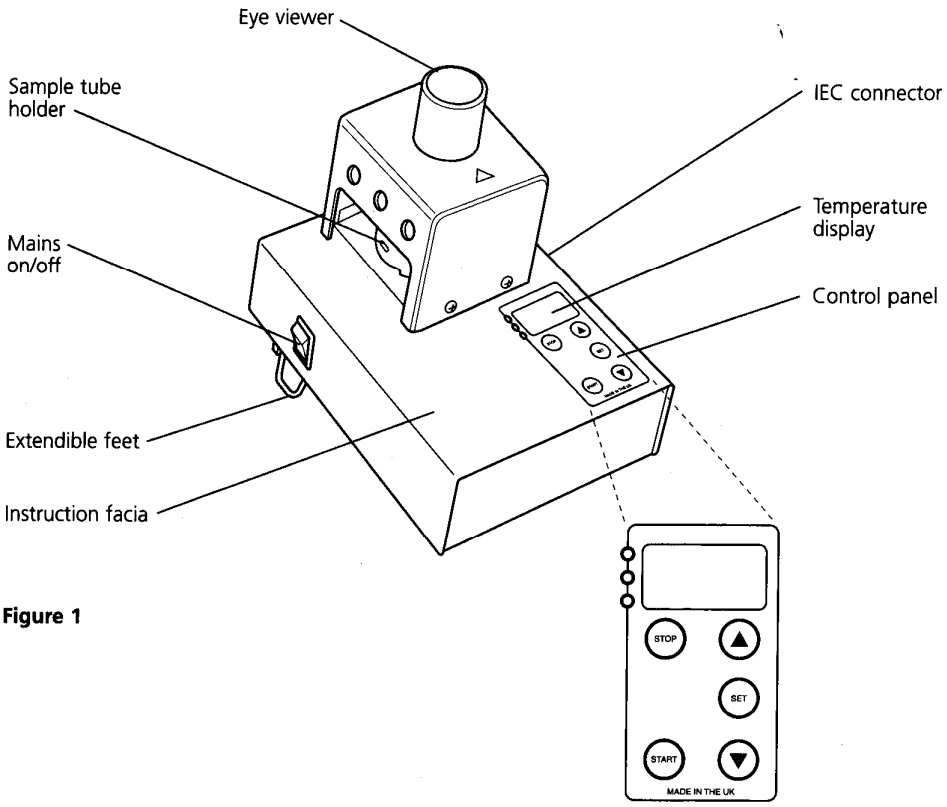



**Figure 1**

### *Use of the Melting Point Apparatus*

1. While the video demonstrated use of the MelTemp, you will use the newer SMP10 melting point apparatus, designed to give quick, accurate results with greater convenience. The SMP has a "plateau" function, which allows you to set a plateau temperature several degrees below the expected melting point. The SMP10 will rapidly heat to this pre-set plateau temperature (~20°C per minute), then hold at that temperature until you're ready to observe the melt. Once your measurement begins, the SMP10 will heat more slowly (~2°C per minute) from the plateau temperature.
2. To use the SMP10, first check to see that all three function lights (STOP, START, and SET) are *NOT* illuminated (See Figure 1). If a light is showing, press the STOP button.
3. Press and *hold* the SET button. The plateau light will flash, and the display will show the plateau temperature set by the previous user. Set your desired plateau temperature by using the arrow keys to scroll the display temperature up or down.
4. *Release* the SET button. The new plateau temperature is now set, and all function lights should go out. (The plateau temperature setting can be checked at any time during operation by pressing and holding the SET button. This action will not interfere with the unit's operation.)
5. Insert the glass capillary tube containing the sample into the side of the heating block via the holes provided. This can be done from either side of the block, which has room to view two samples at the same time.
6. Press the START button. The unit will quickly heat to the plateau temperature. (The *heating light* should be illuminated.) When the plateau temperature is reached, the *plateau light* will *also* become illuminated. There may be some overshoot, so you must wait until the plateau light comes on before proceeding. This ensures that the temperature has fully stabilized at the pre-set plateau value.
7. Press the START button again. The heating block will now begin to heat at the ramp rate of 2°C per minute. (The plateau light should go out, and the ramping and heating lights should *both* be illuminated.)
8. Closely observe the sample throughout the entire melting process, recording the temperature *range* over which the *first crystal begins to melt* and the *last crystal is completely melted.*
9. Press the STOP button. All function lights should go out, and the unit will begin cooling to ambient temperature. Pressing the START button again will cause the unit to return to the plateau temperature (instead of ambient temperature).
10. Remove the glass capillary from the heating block, and discard it in the waste container that is provided.