MELTING POINT

Purpose

This experiment demonstrates the procedure for determining the melting point range of a solid compound using a Mel-Temp or DigiMelt apparatus. This physical measurement can be used as an aid in the identification of a solid substance and as an indication of its purity. The procedure includes identification of an unknown substance using a mixture melting point with a known substance.

Procedure

Melting Point Range

1. Obtain a melting point apparatus. If you are using a thermometer (Mel-Temp), examine the thermometer for air spaces in the enclosed liquid. If there are any, obtain a new thermometer.

2. Obtain small samples of any two compounds whose melting points are *listed consecutively* in the table below. Do not choose the first two or last two on the list. ***Use a clean spatula for each substance – do no contaminate the sample jars or your samples.*** If necessary, grind the sample to a powder.

   Load a small amount of a sample into a capillary tube (1-2 mm) and place it in the slot on the melting point apparatus. Choose an intermediate voltage and observe the rate of heating – if the substance has a relatively low melting point, choose a slower heating rate; if the substance has a relatively high melting point, choose a higher heating rate. As you approach within 10 degrees of the literature melting point, slow down the rate of heating to 1-2 $^{\circ}$C. If the temperature of the melting point apparatus rises faster than the thermometer records the temperature, the recorded melting point will be inaccurate.

   If you are using the DigiMelt, record the starting temperatures and ramp rates you used in your determinations.

   Record the melting point range for the compound, from the time the first droplet of liquid appears until all solid material has become a clear liquid. If the range is wider than 3 degrees, repeat the procedure with a new capillary, using a slower rate of heating. Capillaries cannot be reused. Put used capillaries in the broken glass container.

   In addition to recording data in your notebook, comment on the correspondence between the experimental melting point ranges of your chosen knowns and the listed melting points in the table below.

   ***NOTE**: The thermometer is very likely to be inaccurate in some part(s) of its range. It is very important that you note any discrepancies between the literature melting point and your own carefully-determined experimental range. If you observe the same deviation for both your known samples, especially after multiple determinations, you should apply the same deviation to consideration of your unknown.*

3. Use an approximately 1:1 mixture, ***thoroughly mixed*** by crushing them together, of the same two known compounds chosen above, to perform a mixed melting point determination. In this case, don't repeat the procedure if a wide melting point range is observed since this is expected for a mixture. **Comment on your results in your notebook**. By performing this procedure, you should better understand the melting point lowering and broadening of an impure mixture.

4. Obtain an unknown solid compound from the instructor. Write the unknown's label number in your notebook. The unknown will be one of the compounds listed in Table 1. Determine its melting point range. Narrow down the list of possibilities for the identity of your unknown and state what these possibilities are. Then prove the identity of your unknown compound using the method of mixed melting points. **Explicitly state your conclusions with regard to the results from the mixed melting point**.

If you think there is more than one known compound that could possibly be your unknown, perform mixed melting points on more than one 1:1 mixture simultaneously.

As part of your notebook Results, prepare a table similar to the one on the Report Form (found at the back of this Manual) and enter the appropriate results so they can be easily found and viewed. (Results should also be part of the Observations.)

FIG. 1 A Mel-Temp™ apparatus

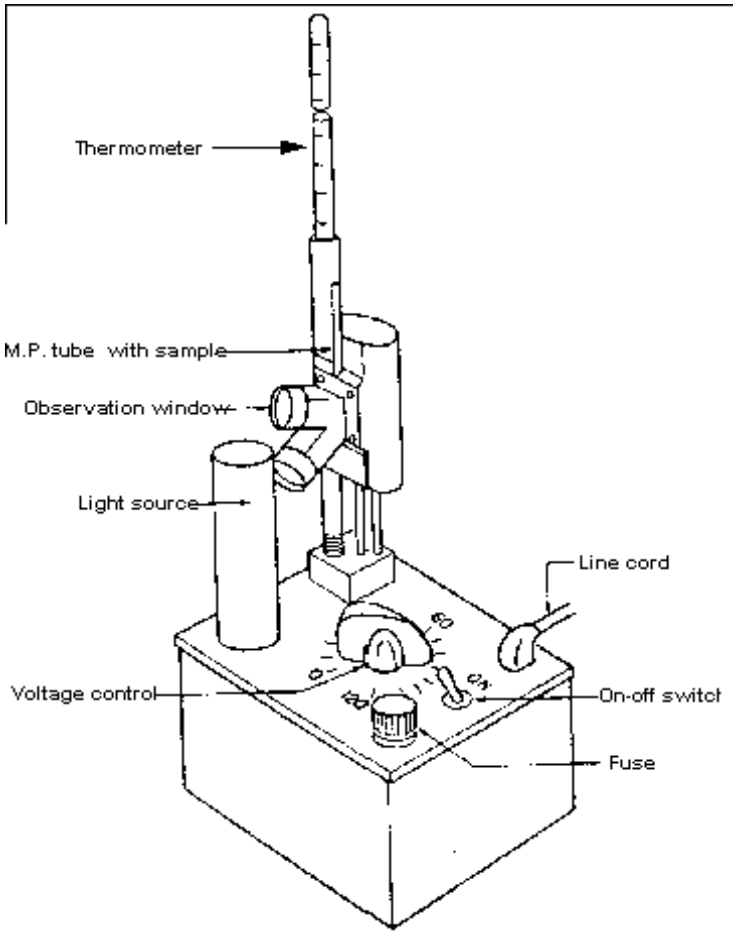

TABLE 1 Possible compounds for melting point unknowns*

Compound	Approx. m.pt. (°C)	Compound	Approx. m.pt. (°C)
acetanilide	115	benzoin	137
d,l-mandelic acid	118	*o*-chlorobenzoic acid	140
benzoic acid	122	anthranilic acid	146
benzamide	128	benzilic acid	150
urea	132	adipic acid	153
trans-cinnamic acid	133	salicylic acid	158
p-acetophenetidide (phenacetin)	135	benzanilide	161

**** Do not use this table of approximate melting points as the literature source you cite in the Citations section of your Report Form. Find the m.pt. in an approved source.***