EXPERIMENT 1: MELTING POINTS

Identity and Purity of Solid Organic Compounds

النقطة الأساسية هنا هي أن درجة الانصهار لا تتأثر بشكل كبير بتغيرات الضغط الخارجي. ببساطة، حتى لو تغير الضغط حول المادة، يظل مستوى الحرارة الذي تبدأ عنده المادة بالتحول من الحالة الصلبة إلى السائلة تقريبًا كما هو. هذا يعني أن الضغط ليس له تأثير كبير على درجة الانصهار في معظم الحالات.

INTRODUCTION

The melting point of a solid is the temperature at which transition fromsolid to liquid occurs at atmospheric pressure; or the temperature at which solid and liquid phases are in equilibrium at a pressure of one atmosphere. The melting point is practically unaffected by changes in external pressure, making it a convenient physical constant for the identification of solids.

Many organic compounds are solids at room temperature as a result of strong intermolecular forces which hold the individual molecules together in a crystal lattice. The nature and strength of these intermolecular forces are responsible for the observed differences in melting point. In general, if the forces are strong, the melting point will be high, and if they are relatively weak, the melting point will be low.

A pure solid has a sharp melting point and will melt within a narrow range of 1-2 °C. Soluble impurities affect the melting point of a solid in the following manner:

a. Lower the melting point of the substance, with the upper limit considerably below the true melting point. The presence of an impurity in the molten compound, reduces its vapor pressure thus lowering the melting point of the compound. The greater the amount of impurity, the greater is the melting point depression.

b. Broaden the melting point range. Depending on the amount of impurity, the melting process may extend over a range of 2-20 °C or more. Insoluble impurities (*e.g.*, glass, sand ...*etc*.) do not affect the melting point or the melting point range.

1 Soluble impurities:
Lower melting point
Broaden range

2 Pure compound:
Sharp melting point
Range 1–2°C

3 Insoluble impurities:
No effect

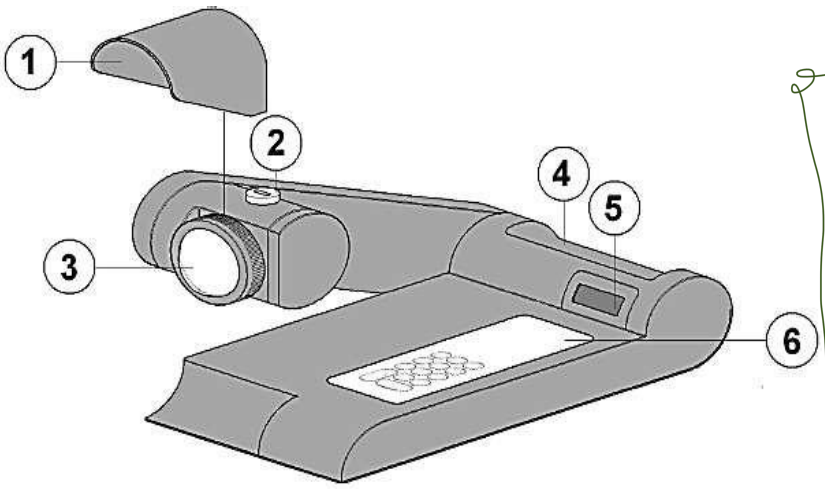
Mixture melting points can be used in the following manner to determine whether two compounds are the same or different even though they have similar melting points. If a given organic compound (A) meltssharply at 120 °C, and benzoic acid (compound B) also has a melting pointof 120 °C. Is compound (A) benzoic acid or a different compound?

If compound (A) is benzoic acid, then a mixture melting point of (A) and (B) will melt sharply at 120 °C, i.e., the same as each individual compound alone. If, on the other hand, compound (A) is not benzoic acid, then the mixture melting point of (A) and (B) will be lowered and the melting range will be broadened. Since they are different compounds, each behaves as an impurity in the other.

MELTING POINT DETERMINATION PROCEDURES

➢ *USING DIGITAL MELTING POINT APPARATUS*

Digital Melting Point Apparatus has been designed for general purpose laboratory use in which samples submitted for analysis are enclosed in a glass capillary tube and brought to a melting point condition under strict controlled parameters of time and temperature. Figure 7 shows a graphical representation for the digital melting point and its components.



Number	Description
1	Lens hood
2	Capillary Guide
3	Lens
4	Capillary tube Holder
5	Digit display
6	Keypad

Figure 7. digital melting point apparatus

GENERAL PROCEDURE

1. The digital melting point apparatus units must always be kept upright.
2. Place a small quantity (about 0.5 cm in tube) of the solid to be melted in a capillary tube (labeled melting point tubes).
3. Tap the closed end of the tube on the desk, clean the outside, and compact the solid down to the closed end of the melting point capillary tube.
4. Drop the tube (closed end down) down a section of glass tubing to compact the solid in the bottom or closed end of the tube even more.
5. Place the tube loaded with the sample into the sample holder of the apparatus with the closed end down. The crystals can be ground up if they are too big to fit into the capillary tube.
6. Melting point capillary tubes are placed (closed end down) in the slots directly in front of the magnifying lens where they are viewed during melting. (Up to three samples can be viewed at once).
7. Record the temperature that the crystals begin to melt; crystals will look wet, (this is the melting start point), and the temperature at which the substance becomes a clear liquid; no solid material remaining (this is the melting end point).

Start point:
crystals look wet
——
End point:
clear liquid

8. Determine the melting range (Starting point – End point).
9. Calculate the melting point by taking the average point between the start point and the end point.

$$\frac{\text{Start's end}}{2}$$

Note

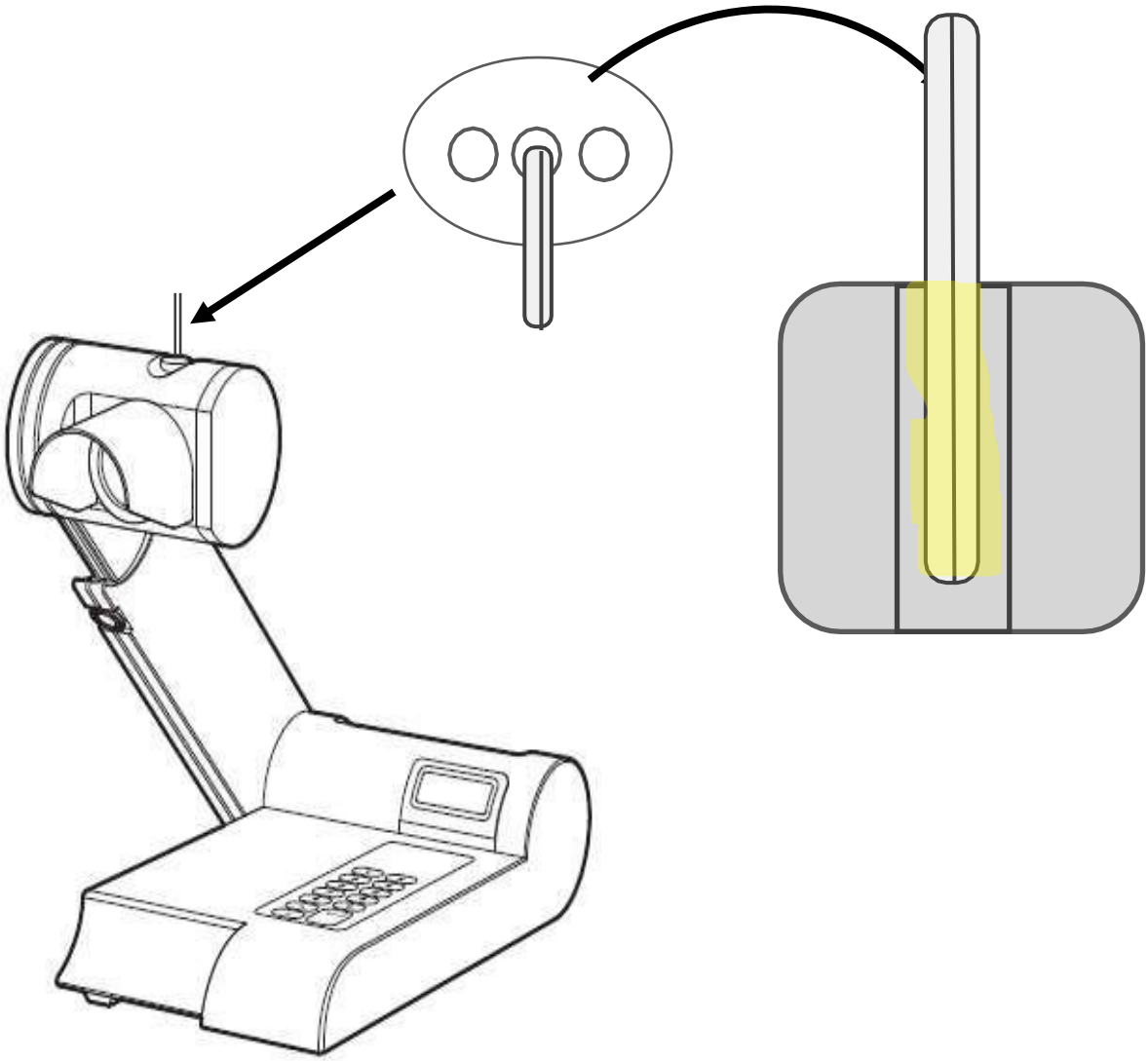
The heating rate of the digital apparatus is adjusted by setting a temperature ramp along with a start and end temperature following the "Quick Start Instructions" on the front of the digital apparatus. A ramp of 20 °C per minute will result in a rapid temperature rise while a ramp of say 2 °C per minute will give a slower rise that will more accurately measure the melting range of a solid.

Slow heating → accurate melting point
Fast heating → inaccurate melting point

OBJECTIVES

1. Determining the melting point of a pure known organic solid.
2. Identifying an unknown from its melting point.

Having done this experiment, you will have seen the effect of an impurity on the melting point of a solid substance and the use of the melting point in characterizing organic solids.



EXPERIMENTAL

MATERIALS NEEDED	Equipment: digital melting point apparatus Glassware: Capillary tubes (open one side only) Chemicals: Binzillide, Salicylic acid, Citric acid, Paracetamol, Caffeine, Urea.

DETERMINATION OF MELTING POINTS OF PURE COMPOUNDS

Each group should obtain a small amount (about 0.1 g) of one of the following solid compounds:

1. Cinnamic acid	2. Salicylic acid	3. Citric acid.
4. Paracetamol	5. Caffeine	6. Urea

Measure the melting point by using the digital Melting point apparatus but you must first measure its melting range.

1. Tap a small amount of your unknown into two different capillary tubes. Just a few crystals are adequate. You may need to grind some of your unknown into a powder if it is too coarse to fit into the capillary tube.
2. Find the melting point range of the pure unknown substance by first quickly determining an approximate melting range on a fast ramp (20 ºC/min from 70-210 ºC)
3. Conduct a slow, careful melting range with the second capillary tube you prepared (use a ramp of 2 ºC/min and start about 15 ºC below the melting range to 10 ºC above the range). Make sure the Digital Melting apparatus is below 70 ºC before starting the first melting range and 10-20 ºC below the compound's melting range before doing a slow careful melting range.
4. After determining the range, now you can calculate the approximate melting point midpoint as follows:

Melting Point Range = (Start Point → End Point)

Melting point Midpoint= (Start Point + End Point)/ 2

IDENTIFICATION OF AN UNKNOWN

Obtain an unknown (from instructor) and determine its melting point as described before. Using the melting points listed in the table on the previous page determine which possible compounds are within ±10 °C of your unknown's melting range.

Table 1. Melting points of some organic compounds

Compound	mp (°C)	Compound	mp (°C)
Acetanilide	114	Maleic acid	135
Mandelic acid	117	Adipic acid	152
2-Naphthol	121	Citric acid	154
Benzoic acid	122	Salicylic acid	158
Urea	132	Benzanilide	161
Cinnamic acid	133	Sulfanilamide	165
Benzoin	133	p-Toluic acid	182

USEFUL LINKS

Melting point of an organic compound-Oil bath method
https://www.youtube.com/watch?v=nQNaTfqXECk

Mixed melting point-melting point apparatus
https://www.youtube.com/watch?v=cLHdm8wJJIw

EXPERIMENT 1
MELTING POINTS
Report Sheet

Name		Section	

OBJECTIVES:

- **Determination of Melting Points of an Unknown.**

Unknown ID	Start	End	m.p Range	Midpoint
	بداية الذوبان	نهاية	end – start	start + end / 2

Your Unknown is expected to be:

- **Look at the following melting point ranges and tick the box to show whether the substance is likely to be a mixture or a pure substance.**

narrow range 1-2 ← | → wide range 2-3 أكثر

Substance	Starts melting (°C)	Finishing melting (°C)	Pure	Mixture
A	**99.1**	**100.4**	✓	
B	**0**	**9.1**		✓
C	**132.2**	**132.4**	✓	
D	**188.2**	**183.8**		✓

- **Two samples have the exact same melting points. Are they the same compound? How could you tell for sure?**

- Not necessarily
  - How to tell for sure:
  - Use Mixture Melting Point: mix the two samples and measure melting point
  - If mixture melts sharply at same point → same compound
  - If mixture melts lower / broader range → different compound