Part 5:
Fundamentals of Preparative Organic Chemistry

CHAPTER-13

Experiment: 13

DETERMINATION OF MELTING POINT OF GIVEN ORGANIC COMPOUNDS

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Aim:

Determination of Melting point of given organic compounds.

Requirements:

A. Glassware & Instruments:

1. Thermometer
2. Melting point capillary tube
3. Spatula
4. Melting point apparatus

B. Chemicals & Reagents:

1. Aspirin
2. Benzoic acid
3. Salicylic acid
4. Distilled water

Theory:

The physical properties of a compound, such as its melting point and boiling point, can be used to identify a sample and determine its purity. These pages describe two common techniques for determining the melting point using a Melting point apparatus and a Thiele tube setup.

The melting point is the temperature at which a solid melts and becomes a liquid. Since this requires that the intermolecular forces that hold the solid together be overcome, the melting temperature will depend on the structure of the molecule involved, illustrating the relationship between structure and properties. Consequently, different compounds have varying melting points.

A pure, nonionic, crystalline organic compound has a characteristically sharp melting point (normally between 0.5 and 1.0 degrees Celsius). A mixture of extremely minute quantities of miscible impurities will lower the melting point and increase the melting point range. Consequently, the melting point of a substance serves as a purity and identification criterion.

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The melting point of an organic solid can be determined by placing a small amount of the solid in a small capillary tube, affixing the tube to the stem of a thermometer centred in a heating bath, heating the bath slowly, and observing the temperatures at which melting begins and ends. Pure samples typically have sharp melting points, such as 148.5-149 °C or 168-169 °C; impure samples of the same compounds melt at lower temperatures and over a larger temperature range, such as 135-138 °C or 196-199 °C.

In order to make the most efficient use of time, it is customary to conduct a rapid melting point determination initially (by heating rapidly) to establish an approximate melting point, followed by at least two additional careful determinations (by heating more gently, i.e. temperature change of no more than 2 °C/min) until two consistent values are obtained.

Generally, the sample is heated indirectly by placing the prepared sample (packaged in a glass capillary or on a glass cover slip) in or on a heated medium, which is most commonly a heated metal block such as a Mel-Temp apparatus. There are various alternatives to the Fisher-Johns apparatus. The Thiele tube method, in which a capillary is immersed in a heated oil bath, is a more basic but equally effective technique. Note that the Thiele tube system is also used to determine the boiling point.

Purpose:

The objective of this experiment is to determine the melting points of numerous organic compounds and use them to identify unknowns.

Procedure:

1. Connect the Melting point apparatus to an electrical outlet and verify that the unit is in the off position.
2. Transfer a small amount of the powder to a weighing boat or paper using a spatula. Fill the capillary tube to a depth of 1-2 mm with an organic compound. (**Note-** The use of large samples runs the risk of producing results with extremely large errors)
3. Insert the tube's open end into the compound. Now, a portion of the sample will be at the top of the tube. Tap the rounded bottom of the capillary tube on the lab table. The sample should then fall to the bottom of the tube. Proceed to step 4 if the sample does not fall to the bottom of the tube.

4. Tap the bottom of the capillary tube on the benchtop to compress the sample, or drop the capillary tube through the plastic sleeve onto the lab bench. The powder will be packed into the bottom of the capillary tube as it bounces on a table.
5. Carefully insert the solid capillary tube into one of the three slots in the Melt Station's heating block. You can slightly tilt the Melt Station in your direction to get a better view of the heating block.
6. Repeat steps 2 to 5 for up to three samples per sample.
7. Power on the device. Turn the control knob on the Melt Station to the Rapid Heat area. When the red LED illuminates, the Melt Station is heating. Rapid Heat will heat your solid sample at a rate greater than 10°C per minute.
8. When the temperature is within 10 °C of the expected melting temperature of a sample, turn the control dial to that temperature, which will reduce the heating rate to 1.5 °C per minute. Observe your sample closely.
9. It is necessary to heat slowly near the melting point in order to distribute heat evenly. If the sample is heated too quickly, melting will occur across a broad temperature range.

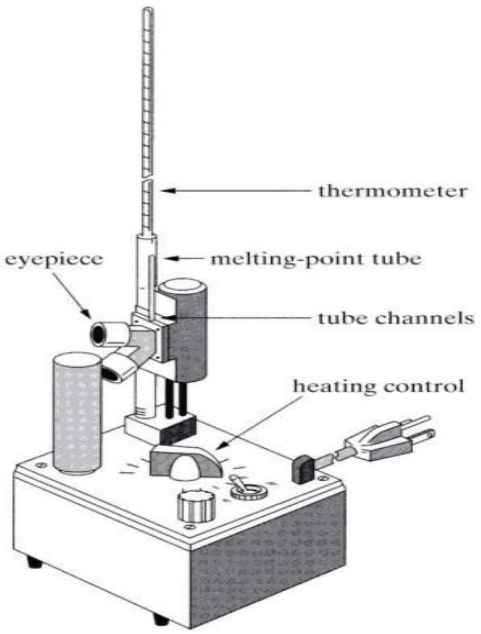

Figure 1 Melting point apparatus

10. Record the melting range, which begins when the sample begins to melt and ends when the sample has melted completely.
11. Once all sample melting ranges have been recorded, turn the control knob on the Melt Station to the Fan/Cooling setting or power off the device. When the blue LED illuminates, the Melt Station is cooling.
12. Prepare additional test samples by repeating steps 3-5. Observe the heating block's temperature on the meter's display. Place new samples in the Melt Station and repeat the melting point testing procedure as necessary once the heating block has cooled to an appropriate temperature.
13. Input your information into the experimental data table. Refer to the table of references to identify the unknown (s).

Calculations:

Experiment Results		
Sample	**Melting Point Range (°C)**	**Compound Identity**
Aspirin		
Benzoic acid		
Salicylic acid		
Benzoin		

Reference Table		
Sample	**Melting Point Range (°C)**	**Compound Identity**
Aspirin	138-140	
Benzoic acid	121-123	
Salicylic acid	158 - 160	
Benzoin	134 - 136	

Applications:

1. **Purity:** The sharp melting point of an organic compound indicates its high purity. A laboratory experiment involving the determination of melting point will shed light on the concept of purity.

2. **Identity:** A definite and sharp melting point is an identifying characteristic of pure crystalline organic compounds. The determination of melting point is a valuable identification criterion for organic compounds.

Result:

Melting point of given sample of organic compound is ..... Melting point of given sample of organic compound as per I.P. is…….

Conclusion:

If the melting point of a given organic compound differs from the value listed in an official book or publication, it may be due to impurities in the given organic compound. Therefore, the provided sample is (pure/impure).

Viva questions:

- Describe the "melting point" of a material.
- Why is it necessary to determine melting points?
- Why isn't this technique used to determine the melting points of inorganic substances?
- Why could the heating rate impact the melting point?
- Define melting point
- What role does melting point play in the identification of organic compounds?
- Whether impurities raise, lower, or have no effect on the melting point.
- What other liquid can be utilised if liquid paraffin is unavailable?
- Why is slow heating necessary to obtain an accurate melting point?
- Why is it necessary to use a calibrated thermometer to determine the melting point?
- What is the range of the organic compound's melting point?

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