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Research Article

Identification of an Unknown Organic Compound by Determination of its Melting Point

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***Abstract*—**the melting point is very useful information for identifying an unidentified organic substance. It is also helpful to define the purity of the substance. Pure substances have a melting point range of 1-2ºC, while the impurities lower the melting point and increase the melting range. In a mixture of two substances, the melting point can be calculated by creating a two-phase melting diagram. The lowest temperature at which the melting point of a mixture can be reduced is called the eutectic point. The melting point is measured on a special heated apparatus using a glass capillary tube. The sample is placed in the tube, which is heated in the device. In the following study, the melting point of an unknown organic compound was measured by the fast and the slow rate heating method. The melting point of the sample showed that the chemical compound to be identified was either acetanilide or 4-nitrophenol. Equal amounts of the unknown substance and acetanilide were mixed, and the melting point was estimated for the mixture. If the mixture has a lower melting point compared to that of the unknown compound, then the two compounds are different, and one acts as an impurity to the other. The mixture had a similar melting point to that of the unknown compound, and therefore the unknown compound was identified as acetanilide $C_6H_5NH(COCH_3)$.

Keywords— melting point range, eutectic, two-phase diagram, mixed melting point, identification, purity

1. Introduction

The melting point is used not only for identifying an unknown organic substance but also for determining the degree of its purity. The melting point was the primary indicator of purity for solid organic compounds until the development of recent spectroscopy and chromatography. The procedure involves slowly heating a small sample of the substance in a melting point apparatus, which is fitted with a thermometer and a magnifying glass for detailed observation of the sample. There are two temperatures that have to be considered. The first is the temperature at which the initial droplet of liquid is formed within the crystalline structure, and the second is the temperature at which the entire crystalline mass has melted into a liquid state. These two critical temperatures consist of the melting point range of the substance [1].

The lowest temperature, at which both liquid and solid states can exist together in equilibrium, is called the melting point of a pure compound [2]. Determining the melting point is extremely valuable for the identification of an organic substance. The change from solid phase to liquid state is very abrupt (1-2ºC) and can be affected by the presence of other substances (impurities). For this reason, it is an important criterion for the purity of a substance [3]. Impurities in a substance lead to a decrease in the melting point, and also an increase in the range of temperatures over which melting occurs. Moreover, the presence of impurities reduces the freezing point. The freezing point can be viewed as a cumulative characteristic, representing the melting point (transition from solid to liquid) when approached from the opposite direction (transition from liquid to solid). Insoluble impurities do not affect the melting point of a compound.

Ions or molecules are arranged in a normal geometric way in a solid crystalline substance. The melting process takes place at a temperature at which the thermal energy of the particles is high enough to overcome the intermolecular forces (dipole-dipole interactions, hydrogen bonds, Van der Waals interactions) that maintain the crystalline structure. The melting process disrupts the organized crystalline arrangement. The melting point indicates the balance of intermolecular forces within a crystal. The more symmetrically arranged the crystals of a compound are, the more consistently it is packaged, and the higher its melting point is. Ionian compounds have a higher melting point than non-polar compounds due to stronger ionic bonds [4].

When it comes to a mixture of two compounds A and B, the behaviour during the melting process depends on the relative amounts of A and B. As shown below in the diagram in Figure 1, when the mixture consists only of pure compound A (not B at all), it has a melting point T_A (point A). When it consists only of pure compound B (not A at all), the mixture has a melting point of T_B (point B). However, when the mixture consists of 80% A and 20% B in mole ratio, it has a melting point T_M (point M). The more substance B is added to the mixture, the lower the melting point gets. There is a limit to how much the melting point can be lowered, and this is when the liquid mixture of compounds A and B reaches saturation with substance B. This lowest possible melting temperature (T_E), in which a mixture of A and B remains liquid, is called the eutectic point (point E), and the mixture is called the eutectic mixture (55%A and 45% B).

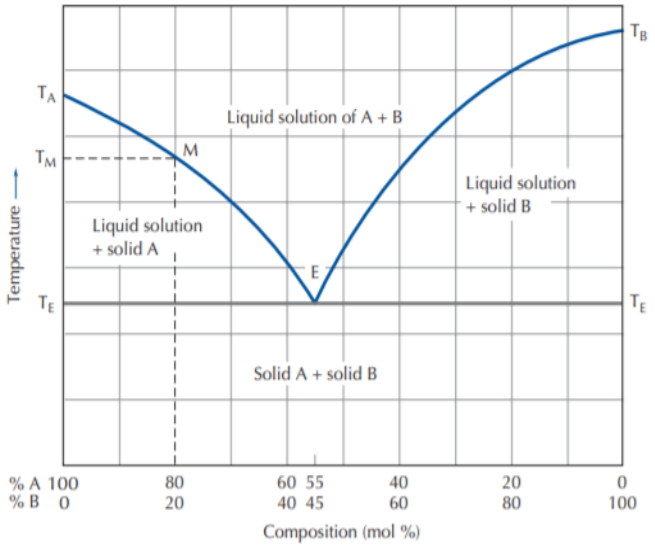

Figure 1: Two-phase melting point diagram of a mixture of substances

A solid mixture of compounds A and B with a eutectic composition is melting, without changing the composition, at a certain temperature (not a range). For example, a mixture of H_2O and NaCl with a composition of 77% and 23%, respectively, constitutes a eutectic mixture with a melting point of -21.1ºC. For this reason, salt is thrown on icy roads; that is, the eutectic composition is achieved, and the ice with the salt melts at a temperature of -21.1ºC [5].

The accuracy of the melting point presupposes:

- Careful preparation of a sample. Drying, grinding, and homogenization of the sample, if it is a mixture of substances.
- Correct sample quantity. A higher amount requires more heating, resulting in an increased melting range.
- Correct puncture of the sample in the capillary tube.
- Periodic temperature calibration control of the heating device. The calibration is performed by measuring the melting point of a few selected organic compounds as reference compounds. [2]

This study will demonstrate how to determine the melting point of an unknown organic compound and then identify its chemical formula by using this.

In the present paper the first section involves the introduction of the melting point, in the second section related work is presented, the third section explains the experimental procedure for the melting point calculation, in the fourth section the results of the melting point of an unknown organic compound are discussed, and the final fifth section is the conclusion and future scope on the research work of this study.

2. Related Work

The first time melting point range was used as a way to identify an organic compound was by Jean Timmermans, a Belgian physic chemist, in 1832 [6]. Melting point range identification constitutes a primary method to determine the identity and structure of an unknown organic compound [7]. The melting point of organic compounds defines the temperature at which a steady, well-ordered crystalline compound attains thermodynamic equilibrium, thereby transforming from the solid state to the liquid state [8].

The determination of the melting point has to be precise. The precision of the melting point range serves as an effective indicator of the purity of an organic molecule. A narrow melting point range suggests a higher degree of purity in the compound, while a wide melting point range suggests impurities [9], [10]. The determination of the melting point is of great importance for newly synthesized compounds. A precise melting point range might provide the most reliable and useful information [11].

The melting point is closely associated with several significant characteristics of pharmaceutical molecules, particularly solubility, which can be considered the most important physical and chemical characteristic of newly designed drugs, and partition coefficient [8]. Estimating the melting point is essential for the development of new pharmaceuticals with targeted solubility. The determination of the melting point of an organic compound can be achieved by evaluating both the enthalpy of fusion and the entropy of fusion [12].

Over the years, a variety of instruments have been utilized to determine the melting point range of organic substances. Nowadays, the melting range is often measured using specialized heated capillary apparatus. However, this technique carries a risk of calculation errors due to the potential for overheating the sample. Furthermore, the absence of more reliable structural analysis methods, such as mass spectrometry and nuclear magnetic resonance spectroscopy, can result in inaccurate conclusions [13], [14].

3. Experimental Method

A small amount (approximately 0.2 grams) of the solid unknown substance is placed in the mortar. With the help of

the knot, the solid is pulverized, and a small amount of it is transferred to a special capillary glass tube. The capillary glass tube has extra thin walls and an internal diameter of 1mm.

A single capillary tube, which is open at both ends, was used. The tube was closed at one end with a Bunsen burner before the introduction of the sample. The tube must be closed by simple contact with the hot (blue) flame of the Bunsen burner and placed at an angle as shown below in Figure 2. This is mandatory in order to prevent the condensation of moisture in the body of the pipe, which evaporates during the heating process. Special care must be taken in order to not create a glass ball in the closed end due to overheating, as shown in Figure 2, because this may prevent the capillary from entering the measuring device, and will also make it difficult to transfer heat to the sample, leading to errors in the measurement of the melting point.

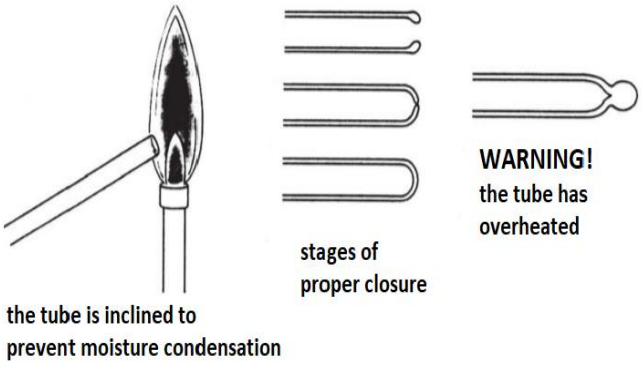

Figure 2: Proper closure of the capillary tube with Bunsen flame

The crystals were introduced into the tube from its open end. The amount of sample entering the tube must correspond to a height not exceeding 1-2mm. It is important that the height of the sample in the glass tube is not higher; otherwise, the melting point range will be unnaturally wide. To transport the crystalline mass to the closed end of the capillary tube, the tube was thrown, with the closed end facing down, inside a glass tube about 0.7m long which was placed perpendicular to the bench. The crystals are gathered to the close end of the tube when the tube strikes the surface of the bench. Additionally, inadequate packing of the solid may lead to shrinkage during heating, potentially leading to confusion regarding the accurate temperature. [15].

NOTICE: It is not recommended to hit the capillary tube on the bench held in the fingers because it is very easy for small pieces of glass to enter the fingers if the tube breaks.

Following the proper packing of the sample into the closed end of the capillary tube, the tube was placed properly in the heating apparatus in order to determine the melting point of the unknown substance. The digital melting point apparatus used in this study, which is shown in Figure 3, is designed to heat the sample at a specific heating rate, which we can adjust as needed, and the sample is observed through a magnifying lens that is part of the apparatus.

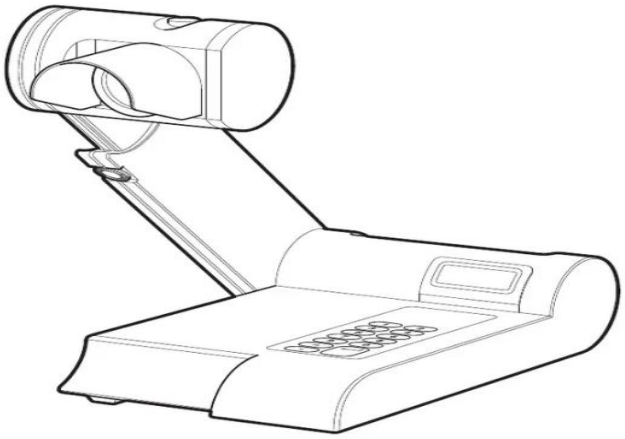

Figure 3: Digital melting point apparatus

During the heating process of the sample, the temperature was recorded twice. The first reading is the temperature at which the first drop of liquid is created between the crystals of the sample, while the second reading indicates the temperature at which the crystal mass of the sample has completely transformed into a transparent liquid. These two temperature readings constitute the melting point range of the unknown organic compound.

The beginning of the melting is considered to be the temperature at which the first drop on the wall of the glass capillary tube is observed. The melting ends after all the solid has settled. The process of measuring the melting point was performed once with a rapid rise in temperature and twice with a slow rise of temperature. In the slow rise in temperature, the heating rate should not exceed 1^{o}C / min. The melting point range of the sample is compared with other known melting points from the literature and the unknown compound is identified.

Once the sample in the tube has been fully melted and the different temperature values, as well as any other observations, have been recorded, is very important that the apparatus is allowed to cool down before measuring the next sample, while the used sample in the capillary glass tube must be carefully disposed of according to the laboratory's instructions for the disposal of sharp objects.

4. Results and Discussion

The initial indication that the sample is approaching its melting point is typically observed as a reduction in the volume of the sample, leading it to retract from the walls of the tube; however, no droplet is formed at this stage. This phenomenon is referred to as the densification of the mass without actual melting. The first droplet appearing between the crystals of the sample becomes noticeable at a few Celsius degrees above the densification point, marking the onset of melting. The melting process is considered complete when the last crystal in the sample disappears. The changes in the crystalline mass of the organic compound during the heating procedure are shown in Figure 4.

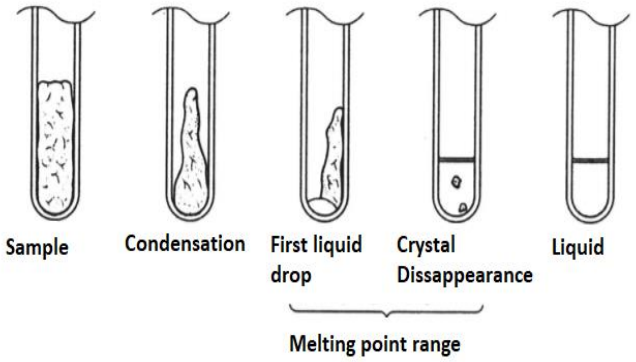

Figure 4: Typical changes in the mass of a substance from solid to liquid

Three separate samples of the unknown organic substance were analyzed, and the melting point range was determined for each sample individually. In the first measurement, the temperature was increased at a fast rate, approximately 10ºC/min, whereas the second and third measurements of the melting point range were conducted at a slower rate, not exceeding 1ºC/min. As shown below in Table 1, the melting point range recorded for the unknown substance in the first measurement was 92-117oC while the temperatures for the second and third measurements were 110-116ºC and 112-116ºC, respectively.

Table 1: Melting point range of the unknown compound With the rapid and slow procedure

Rapid Procedure	Slow Procedure	Slow Procedure
92-117ºC	110-116ºC	112-116ºC

According to the results above, the most accurate determination of the melting point was made in the third measurement; where the temperature range is lower (112-116ºC). Pure substances, as mentioned above, show a sharp change from solid to liquid (1-2ºC). The narrower this range, the clearer the solid compound. The melting point is affected not only by the purity of the substance but also by other factors, such as the size of the crystals and the heating rate of the apparatus. Also, the large range at the melting point may be due to the fact that there was a sufficient amount of the substance in the capillary tube. We should not put a large number of crystals because, on the one hand, as they melt, they shake up a part of the crystals and make it difficult to measure, and on the other hand, the heat is slow to be transmitted from the heated end of the sample to the bottom. This duration increases if the crystals are less densely packed, meaning that the taller the sample in the capillary tube, the longer the time needed.

The actual melting point range of a substance differs from the reading value of the thermometer because the part of the mercury column outside the heating source does not expand in the same way as the part inside the device (the outside temperature is always lower inside the device, so the elevation of the mercury column appears smaller than the actual one). The error is considered insignificant for melting points up to 100ºC and approximately 3-5ºC for melting points in the range of 200ºC.

The substances that have a similar melting point to that of the unknown substance are acetanilide (113-115ºC) and 4-nitrophenol (113-114ºC). The fundamental characteristics of those two compounds are shown below in Table 2. In order to identify which of the two is the unknown substance, we will perform the determination of the mixed melting point [16]. The mixed melting point requires the use of an original sample from a different source. In this process, the two substances (the original and the unknown) are finely powdered and then blended together. After that, the melting point of the mixture is measured. A reduction in the melting point or a significant increase in the melting range compared to the individual substances indicates that one of the substances acts as an impurity to the other and they are not identical. In contrast, if the melting point remains consistent with that of the pure compounds A and B, it is highly probable that they are identical. [5]

Table 2: Primary properties of Acetanilide and 4-nitrophenol

Name	Chemical Formula	Molar Mass	Melting Point	Density
Acetanilide	$C_6H_5NH(COCH_3)$	135.17 g/mol	113-115°C	1.219 g/cm^3
4-Nitrophenol	$C_6H_5NO_3$	139.11 g/mol	113-114ºC	1.48 g/cm^3

According to the above, an amount of the unknown substance was mixed with an equal amount of acetanilide in order to be identified. Based on the most accurate measurement the unknown substance has a melting point of 112-116ºC. The unknown substance is acetanilide if the melting point remains the same, while if there is a decrease in the melting point, then the substances are not identical, and therefore the unknown substance is 4-nitrophenol.

The melting point of the mixture was determined using the slow method of increasing the temperature. The mixture of acetanilide and the unknown substance presented a melting point range of 113-116ºC. Therefore, because no reduction in the melting point range was observed, the unknown substance was acetanilide.

The chemical structure of acetanilide is shown below in Figure 5.

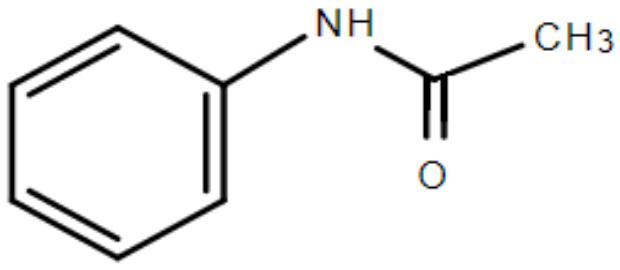

Figure 5: Chemical structure of the substance of acetanilide

5. Conclusion and Future Scope

This study focuses on the identification of an unknown substance that displays a melting point range similar to that of two known compounds, acetaniline and 4-nitrophenol. The precise identification of the unknown compound was achieved by determining the mixed melting point after combining equal amounts of the unknown substance with acetaniline. The precise identification of the unknown compound was achieved by determining the mixed melting point of a mixture contained equal amounts of the unknown substance and acetaniline.

In summary, the melting point serves as a reliable technique for compound identification and also acts as an indicator of its purity. Furthermore, knowledge of the melting point of a compound is essential for the synthesis of pharmaceuticals with specific solubility.

In order to improve the identification process of unknown compounds in future research, the application of nuclear magnetic resonance spectroscopy and mass spectroscopy techniques is recommended.

Data Availability
The data from the experiments conducted in this study can be found in this article.

Conflict of Interest
The author proclaims that has no conflict of interest.

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Authors' Contributions
The author carried out the research, reviewed and edited the manuscript, and approved the final version of the manuscript.

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