

Lab 4 NC

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Advanced Chemistry of OME - ACDA6020

Laboratory Report **Determination of Melting Point**

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Contents

List of Figures	3
List of Tables.....	3
1. Introduction	4
1.1. Aim	4
1.2. Hypothesis	4
1.3. Key Variables.....	4
1.4. Melting Point	4
1.5. Importance of Melting Point Analysis in Energetics	5
2. Results	6
3. Discussion.....	9
3.1. Sources of Experimental Error.....	9
4. Conclusion	11
5. Recommendations.....	12
6. References	13
7. Appendix 1 – Materials and Methodology.....	15
7.1. Materials and equipment	15
7.2. Methodology	15
7.3. Safety.....	16

List of Figures

Figure 1 Melting transition [4].....	5
Figure 2 Sample A – impurities circled.....	6
Figure 3 Sample B – no obvious impurities.....	7
Figure 4 Sample C – no obvious impurities	7
Figure 5 Sample D – impurities circled	8
Figure 6 Cole Parmer melting point microscope [17].....	16

List of Tables

Table 1 Different variables for the experiment	4
Table 2 Melting point temperatures.....	6
Table 3 Sources of experimental error	10

1. Introduction

1.1. Aim

This experiment aims to determine the melting point of four methyl-3-nitrobenzoate samples synthesised in a previous laboratory session, enabling evaluation of the purity of each sample.

1.2. Hypothesis

It is expected the samples will show a degree of variation in their *melting points as a result of different purity.* **purity**. Pure methyl-3-nitrobenzoate will melt between 78 °C - 80 °C, whereas samples containing impurities will have a melting point below the literature value [1] [2].

1.3. Key Variables

For this experiment the variables are shown in Table 1.

Table 1 Different variables for the experiment

Variable	Example
Independent	Four different samples <i>level of purity</i>
Dependent	Observed melting point
Control	Heating rate of melting point microscope
	Sample drying method
	Volume of sample loaded in to capillary tube

1.4. Melting Point

The melting point of a substance is the temperature at which it transitions from a solid to a liquid phase. During transition both phases exist in equilibrium [3]. Pure samples have a sharp melting point, typically with a 1-2 °C range of the literature value. Impure samples have a lower melting point and a broader melting range. This difference occurs due to molecules in pure samples being held in a regular crystalline lattice structure, all intermolecular bonds experience the same forces and simultaneously all melt at the same time. Impurities disrupt the lattice structure and molecules experience different intermolecular forces, lowering the melting point and causing much broader melting range – as it takes less energy to break bonds in impure samples [4]. For example, adding salt to ice will lower its melting point [5]. Figure 1 illustrates the process of melting as the temperature of the sample increase, showing melting is complete when all the sample is liquid.

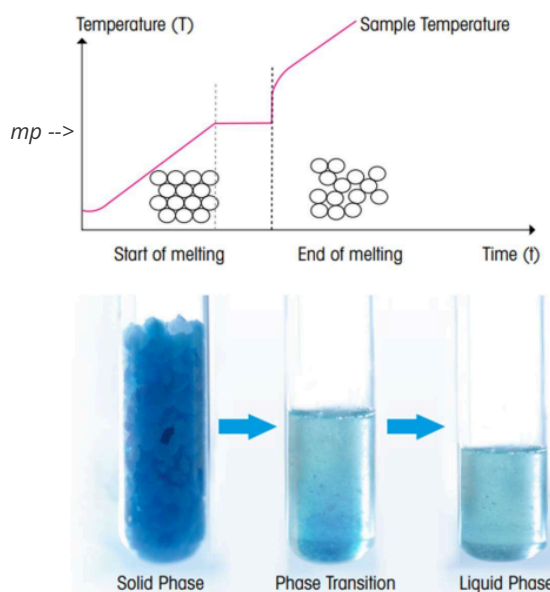


Figure 1 Melting transition [4]

1.5. Importance of Melting Point Analysis in Energetics

Melting point determination is a common technique for assessing purity and characterising crystalline substances, in particular organic material. Measurement is simple and reproducible, providing rapid and reliable analysis. As melting points are sensitive to the presence of impurities, the test is used to assess the purity of substances and identity of materials [6] [7]. Melting point testing is routinely performed when scaling up the production of new energetic material, and during routine manufacture. Understanding the thermal behaviour of energetic material is essential for safe handling with testing providing confirmation of purity and that the material is safe to handle [8] [9]. Melting point testing also has considerable in-service safety implications for storage, use and transport. If an energetic material is heated close to its melting point, its mechanical properties and sensitivity may change. For example, trinitrotoluene (TNT) has a melting range of 80 °C - 82 °C [10]. Poor storage conditions or leaving an item containing TNT in the barrel of a gun in direct sunlight, could result in the item approaching this temperature, partially melting and subsequently affecting performance and safety. Therefore, STANAG 4170 and AOP-7 both require melting point to be determined during characterisation of explosives [11] [12].

2. Results

The observed melting points for all samples are shown in Table 2 and compared literature values [1] [2]. Supporting images are shown in Figure 2 to Figure 5, with examples of impurities highlighted.

Table 2 Melting point temperatures

Sample	Appearance	Melting point			Difference from literature - 78 °C (°C)
		Trial 1 (°C)	Trial 2 (°C)	Average (°C)	
A	Pale orange, clumpy	72	71	71.5	-6.5
B	White, fluffy, small crystals	77	77	77	-1
C	Slightly yellow, fluffy, small crystals	77	77	77	-1
D	Slightly yellow, fluffy, small crystals	73	74	73.5	-4.5

you should have recorded the range of temperatures from the first signs of melting till all sample turned into liquid



Figure 2 Sample A – impurities circled



Figure 3 Sample B – no obvious impurities

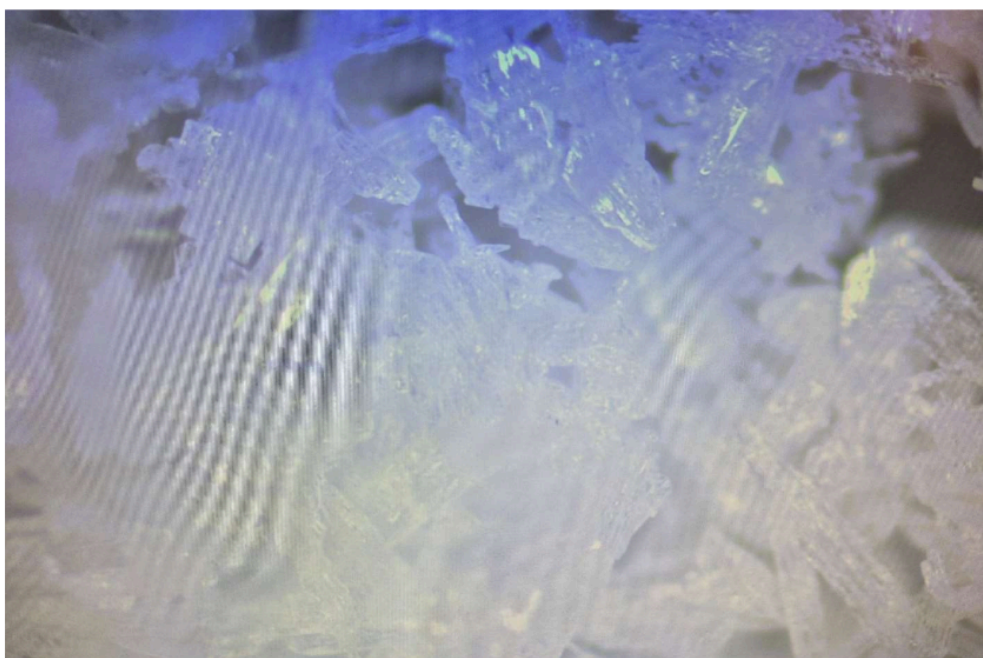


Figure 4 Sample C – no obvious impurities



Figure 5 Sample D – impurities circled

3. Discussion

Samples B and C had a melting point one degree below the literature value for methyl-3-nitrobenzoate with identical repeat measurements. This consistency indicates both samples are of high purity, aligning with the hypothesis. In contrast, samples A and D showed a much lower melting point and slight variation in repeated measurements, which is characteristic of the presence of impurities and what would be expected, as highlighted in the hypothesis.

All samples were examined under a Brunel BM-XFCAM1080 microscope, allowing microscopic assessment of the samples. Figure 2 (sample A) and Figure 5 (sample D) show dark areas (circled), which are indicative of impurities, supporting the lower melting point of the two samples.

Visual examination of the samples provided information on colour and texture, noted in Table 2. Methyl-3-nitrobenzoate is typically white, cream or pale yellow [13]. Sample A appeared orange, different from literature descriptions, indicating it might have a lower melting point than literature values. Sample B appeared white, and samples B and C were slightly yellow. White to yellow colouring is to be expected and would not indicate contamination at this stage.

Figure 3 (sample B) and Figure 4 (sample C) do not show any obvious impurities. While this does not confirm the samples are completely pure, the absence of visible impurities, the measured melting points, and consistent results do indicate high purity samples.

3.1. Sources of Experimental Error

Primary sources of error are identified in Table 3. These are heating rate, sample loading and visual observation of melting point. Mitigation factors are provided for these points; several sources of secondary errors are also identified.

Table 3 Sources of experimental error

Primary source of error	Problem	Solution	Example
Heating rate	A high heating rate can cause the sample temperature to lag behind the thermometer, leading to artificially high measurements	Use a slow, controlled heating rate, particularly around the expected melting point	2-3 degrees per minute
Loading of capillary tube	Inconsistent packing of sample into capillary tube - uneven sample or gaps leading to uneven heat transfer and inconsistent melting temperature	Consistent loading method and consistent volume loaded into tube	2mm of sample in the tube
Visual observation of phase change	Subjective observation of melting point, identification of melting point may vary between users leading to variability	Use an automated melting point microscope which detects changes in light transmission to reduce observer bias	Mettler Toledo Melting Point System MP30
		Perform repeat measurements to improve consistency	At least five repeats
Secondary source of error	Problem	Solution	Example
Sample dryness	Residual moisture or solvents can lower and broaden the melting point	Ensure sample is fully dry before testing, store sample correctly prior to testing to avoid moisture ingress	Buchner funnel, dessicator, vacuum oven drying
Out of range thermometer	Onboard equipment thermometer may be out of calibration and not read the correct temperature. This may simply be due to age of equipment or natural drift	Perform a baseline check with a sample of known melting point, to ensure equipment is within specification - use a sample with a similar melting to those expected in testing	Sample are test dependent

4. Conclusion

The experiment determined the melting point of four samples of methyl-3-nitrobenzoate. A single repeat was performed for each sample. The average melting point for each sample was:

A – 71.5 °C

B – 77 °C

C – 77 °C

D – 73.5 °C

Observation of samples under the digital microscope supported the melting point data. Sample A and D had lower melting points, indicating the presence of impurities. In contrast, samples B and C were found to be purer, both with a melting point only one degree from the literature value with observation under the microscope showing no obvious impurities.

Melting point microscopy is a quick and simple test, providing an indication of purity. However, the technique can be strengthened when combined with other methods and by following best practice guidelines for laboratory work, such as performing a suitable number of repeats. This is discussed in more detail in Recommendations below.

5. Recommendations

Given the quantity of material available of the samples, further work for this experiment should initially focus meticulous sample preparation, drying samples overnight in a desiccator as moisture can lower melting points; and increasing the number of repeats for each sample to improve confidence in the results.

Using an automated melting point microscope, such as the Mettler Toledo MP30 noted in Table 3, reduces observer bias during phase change. Complimentary methods, such as Differential Scanning Calorimetry, could determine the melting point and analyse the purity of samples more objectively as data is produced rather than the relying on the observer [14] [15]. Thermogravimetric Analysis measures the moisture content of samples, providing more detailed results [16]. Thorough sample analysis is important for energetic materials to allow confidence in the results for safe handling, use and storage.

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7. Appendix 1 – Materials and Methodology

7.1. Materials and equipment

- Methyl-3-nitrobenzoate
- Cole-Parmer M200 melting point microscope
- Glass capillaries
- Brunel BM-XFCAM1080 microscope
- Glass plates for Brunel BM-XFCAM1080 microscope

7.2. Methodology

Melting point microscope – see Figure 6 below for a labelled image of the microscope:

1. Set up the melting point microscope by placing it on a firm flat surface. Connect it to a power supply and switch on the microscope. The microscope will show the temperature of the block, and the sample chamber light will activate.
2. Set the plateau temperature on the microscope. To do this, press and hold the “plateau set” button and adjust the temperature with the arrow keys to 10 °C below the expected melting temperature of the material. Release the button to confirm the temperature.
3. To prepare a sample for analysis, take a capillary tube, noting the open end and gently tap this into the sample. A small amount of sample will stick to the tube. Ensure 1-2mm of sample has been collected.
4. Invert the tube so the sealed end is now pointing down, tap it lightly onto the bench. The sample will then slide further into the tube. Repeat this action until the sample is in the centre of the tube.
5. Place the capillary tube in to the sample hole in the block, ensuring it is visible through the lens.
6. Press “start” to quickly heat to the plateau temperature. Once stabilised, the plateau light will turn on.
7. To observe the melting point, press start again and watch the sample through the lens. The temperature will begin to slowly ramp up at 2 °C/minute. Record the temperature when melting is first observed.
8. Press “stop” after observation is completed and allow the device to cool before the next use.

Note – the equipment will cool faster if it is switched off rather than just pressing stop. Both are valid methods, depending upon the time available.

9. Clean the workspace after the experiment is finished, including disposing of any waste.
10. Ensure the melting point microscope is switched off and all samples have been removed.

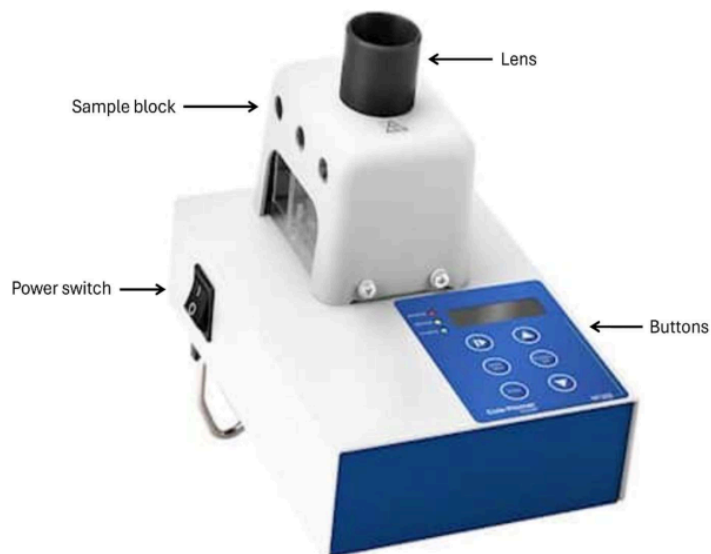


Figure 6 Cole Parmer melting point microscope [17]

Brunel BM-XFCAM1080 microscope:

1. This microscope will already be set up ready for use.
2. Place a small amount of a sample on a glass plate. Label the glass plate with the sample identifier.
3. Place the glass plate on the bed of the microscope, adjust the focus using the dials on the reverse of the microscope to observe the crystals clearly.
4. Move the glass plate slowly to examine other areas of the sample.

7.3. Safety

Appropriate personal protective equipment must be worn:

1. Laboratory coat
2. Gloves
3. Googles

Long hair must be tied back.

Clean any spillages immediately and dispose of waste through the correct routes. Ask if this is not clear.

Sharps – glass capillary tubes can break and must be handled with care. Inform a technician if a breakage does occur and clean fragments with caution. Dispose on any broken glass in the glass waste bin. Seek assistance immediately in case of any cuts.

Burns – the heat block on the microscope will become very hot. The block is isolated; however, samples will be hot when removed from the equipment. Handle with caution and do not pick up the tube from the centre immediately after removing it from the microscope. Place any burns under cold running water for at least 10 minutes.

FINAL GRADE

78/100

GENERAL COMMENTS

Engagement with Literature 13/15

You have clearly identified the literature melting point for methyl-3-nitrobenzoate and supported it with credible sources. There is an excellent breadth of literature, including textbooks, journal articles, standards, and reputable technical sources. Importantly, the literature is not simply listed but is used effectively to support both theoretical explanation and interpretation of experimental results, including the applied OME context. Referencing is consistently in IEEE style. To reach full marks, you could further prioritise primary journal literature over manufacturer data where possible.

Knowledge and Understanding 16/20

The aim of the experiment is clearly stated, and your understanding of melting point behaviour is strong. You provide a clear definition of melting point and correctly explain why pure compounds melt sharply, using disruption of the crystal lattice and intermolecular forces to explain melting point depression and range broadening in impure samples. Chemical language is used accurately throughout.

Variables are mostly well identified; however, the independent variable would benefit from being defined more explicitly in terms of degree of purity rather than simply different samples. This refinement would strengthen the scientific framing of the experiment.

Cognitive and Intellectual Skills 16/20

A clear and appropriate hypothesis is presented. You demonstrate strong analytical thinking by comparing experimental values with literature data and linking melting behaviour to crystal appearance. The use of supporting examples (e.g. ice-salt systems and energetic materials) strengthens your reasoning. Results are interpreted rather than merely described,

and conclusions are explicitly linked back to the hypothesis.

Further depth could be achieved by placing greater emphasis on melting ranges rather than midpoints alone.

Practical Application Skills 21/30

The experimental methodology is clearly described and appropriately placed in the appendix. You recognise the importance of heating rate control and demonstrate good awareness of experimental uncertainty, including observer subjectivity. Potential sources of error are well analysed, with sensible mitigation strategies proposed, and suitable alternative analytical techniques are suggested.

However, only melting point midpoints are reported, rather than full melting ranges, which limits the strength of purity assessment. Including ranges would significantly improve the robustness of the analysis.

Transferable Skills 12/15

The report is clearly and professionally structured, with an excellent word count. Tables and figures are well presented and clearly captioned, and the overall standard of written communication is appropriate for Level 6 scientific reporting.

Minor improvements could be made through tighter concision in places, but overall presentation is strong.

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PAGE 15

PAGE 16

PAGE 17
