

ACDA6020 Lab Session 4: Methyl 3 Nitrobenzoate

Melting point

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Introduction

The aim of this investigation is to obtain the melting point of the student synthesised Methyl 3 nitrobenzoate samples to evaluate the purity of the samples. These samples were synthesised in lab report 3. The samples which are purer will be within a closer range to the stated melting point. The stated melting point of Methyl 3 Nitrobenzoate is 78-80 degrees [1]. 4 samples are being used within this report from 4 pairs of students.

Variables

- Independent: The different samples.
- Dependent: The observed melting point temperature.
- Controlled: Ramp rate (2°C/min), plateau temperature setting, and the specific apparatus (Cole-Parmer M200).

Theory

Key theory used in this investigation is the melting point of a material. The melting point is the temperature where the material changes state from a solid to a liquid [2]. The melting point is used to determine the purity of the sample. This is due to the Structure of the substance, a pure chemical like MNB the molecules pack densely and uniformed lattice, this means that all

the bonds break at the same temperature resulting in a tight melting point usually. When a substance is contaminated, the molecules aren't able to pack densely or uniformly, due to this the forces holding the molecules together is weaker resulting in a lower melting point.

Purity of samples was dictated in the synthesis process during the recrystallization step. The samples were recrystallized using hot ethanol. Ethanol is used because it can dissolve the methyl 3 Nitrobenzoate when it is hot and the contaminants, however when the ethanol cools the methyl 3 nitrobenzoate is not soluble by ethanol leaving the product behind and removing contaminants.

Results

The melting point of methyl 3 nitrobenzoate is 78-80 degree Celsius as previously stated [1].

Group	Inspection	Trial 1 MP	Trial 2 MP	Average MP
DG	Clumpy	77	77	77
OM	White	75	75	75
LM	Odour, yellowing	74	73	73.5
B J	Clear	77	77	77

DG and B + J samples were the closest to the stated melting point of MNB being just outside by 1 degree. With LM being the furthest from the melting point 4.5 degree away. DG was stated to be clumpy which suggests a texture difference but not necessarily an impurity. LM was observed to have an odour and a yellowing which deviates from the stated description. OM sample is the closest to the stated sample.

Discussion

DG and B + J had a MP of 77 degrees is the closest to the stated MP of MNB suggesting they could be the highest percent purity samples. There is evidence to suggest impurity within the LM sample due to the low melting point of the sample at just 73.5 degrees average. This is due to the impurities affecting structure of the sample. Adding to this evidence the odour and yellow stain suggests that the sample is impure. This impurity

may be due to the sample being cooled too rapidly trapping contaminants in the crystals. Another potential reason for contamination could be inadequate drying which could suggest the presence of ethanol contamination.

Errors

The Cole-Parmer M200 used as the method of heating the samples and observing the melting points. The Cole Parmer M200 has a stated accuracy of ± 1 at 20 c and ± 2.5 at 300c [3]. Therefore, at 80c could have discrepancy of 1.5c. This range could exacerbate or minimise our results compared to the stated MP of MNB.

The capillary tubes require being packed correctly to ensure the temperature is transferred to the chemical effectively. The tube should be filled with material about 2-3mm wide [4] and packed tightly. The sample should be completely dry for accurate results.

This experiment is completed by an operator recognising a state change within the capillary tube. This allows for human error to creep into the results. If the sample is contaminated by solvent this will create droplets of liquid in the tube first before the MNB melts. This can create the illusion that the sample is melting this is called sweating. Sintering is another factor which is where the solid molecules contract and can be mistaken for “sagging” creating the illusion of melting.

OME applications

Purity of samples in the OME worlds is critical to product safety and quality. Contamination of an Energetic material decreases the insensitivity of the munition. This reduces the safety of the product and will increase manufacturing, transportation, disposal and firing accidents [5].

Alternatives

Another option of assessing the purity of the sample to confirm the results would be to use FTIR. FTIR works by measuring the absorption of infrared light by chemical bonds. This can be used to check the purity of the sample [6].

Conclusion

The samples of DG and B + J are determined to be the purest by this experiment. The theory of contaminated samples would melt before the stated melting point of MNB. For more confirmation of this data it would be recommended to complete other methods stated above like DSC.

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7. Appendix

Materials:

Cole-Parmer M200

glass capillaries

The methyl-3-nitrobenzoate samples from each group.

ACDA6020 LAB SESSION #4

DETERMINATION OF MELTING POINT

AIM OF THE EXPERIMENT: Determine the melting point of four methyl-3-nitrobenzoate samples synthesised in Cranfield to evaluate the purity.

INTRODUCTION

The melting point of a substance is the temperature at which it changes from a solid to a liquid. It is an important physical property used to help identify a compound and assess its purity. A pure organic solid typically melts over a very narrow temperature range, often within 2–3 °C of its literature value.

The presence of impurities, even in very small quantities, affects how a sample melts. Impure solids usually:

- melt at a lower temperature than the pure compound, and
- show a broader melting range.

Melting begins when the first droplets of liquid appear among the crystals and ends when the sample becomes fully liquid.

Recrystallisation is a common purification method for solid organic compounds. The process involves:

- Dissolving the impure compound in a suitable hot solvent.
- Cooling the solution slowly so that only the pure compound crystallises.
- Separating the pure crystals from the solvent and any remaining impurities.

This technique relies on differences in solubility between the desired product and its impurities: the pure compound crystallises out, while most impurities either stay dissolved or are removed by filtration.

For this experiment, methyl-3-nitrobenzoate was purified by recrystallisation from hot ethanol. This removed impurities such as unreacted starting materials and minor side-products formed during the nitration reaction. As the solution cooled, clean crystals of the product formed and impurities remained in the solvent.

You will measure the melting points of 5 samples of methyl-3-nitrobenzoate synthesised at Cranfield. By comparing the melting behaviour of each sample, you will assess the purity of the final nitration product.

POTENTIAL HAZARD

	Hazards
Health Hazards	Methyl-3-nitrobenzoate • may be harmful if swallowed, inhaled, or absorbed through the skin. • can cause irritation to the skin, eyes, and respiratory tract. • prolonged exposure may lead to sensitisation or allergic reactions in susceptible individuals.
Flammability and Combustibility	While not highly flammable, methyl-3-nitrobenzoate is combustible and can release toxic fumes, such as carbon monoxide, carbon dioxide, and nitrogen oxides, when burned
Environmental Hazards	It may pose a risk to aquatic environments if released improperly, as nitroaromatic compounds can be toxic to aquatic life.
Reactivity	Under certain conditions, nitro compounds can act as oxidising agents, potentially posing a hazard when in contact with strong reducing agents or combustible materials.

DATA SHEET to RECORD OBSERVATIONS, CALCULATIONS and RESULTS

STUDENT'S NAME: _____

PARTNERS' NAMES: _____

REPORT IS DUE DECEMBER 5TH, 2025

submitted via MOODLE as ONE .doc file

PROCEDURE

Put on the lab coat, safety goggles, and gloves. Make sure that long hair is tied.

IMPORTANT: Record all your observations, calculations, and conclusions in the data sheets at the end of this script.

Lab tools and supplies:

Cole-Parmer M200 Melting Point Microscope (MPM)

Glass capillaries

Samples of methyl-3-nitrobenzoate synthesised and purified in Cranfield.

Operating procedure will be discussed in the lab however the main points are:

1. **Set up the MPM device** by placing it on a stable surface, connecting to power, and switching it on. The display will show the block temperature, and the sample chamber light will activate.
2. **Prepare samples for the analysis** by filling a capillary tube with each sample.
3. **Set Plateau Temperature:**
 - i. Press and hold the 'Plateau Set' button; adjust the temperature with arrow keys to 10°C below the expected melting point.
 - ii. Release the button to confirm the setting.
4. Place the capillary tube into a sample hole in the block and make sure that it is visible through the lens.
5. Press 'Start' to heat quickly to the plateau temperature. The plateau light will turn on once stabilized.
6. **To observe melting point**, press 'Start' again to begin slow ramping (2°C/min). Observe through the lens and record the temperature at the first sign of melting.
7. Press 'Stop' after observation is completed. Allow the device to cool before the next use.

Safety Notes:

- Handle with care; the block becomes very hot and the glass capillaries can break.
- Use correct PPE.

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REPEAT THE MEASUREMENTS TWO TIMES FOR EACH SAMPLE

Literature values of melting points:

	melting point, °C
methyl benzoate	
methyl-3-nitrobenzoate	78-80

Experimental values of melting points

Sample	appearance	trial 1	trial 2
		mp, °C	mp, °C
A		DC LB 76.7	un N 74
B		DC LB 77	un N 73
C		om CH 74.5	SD B 77
D		om 75	SD B 77

OBSERVATIONS AND OTHER RELEVANT INFORMATION

10.1530

DC - clumpy buff white
un + N -