

Lab 4

by Libbie Bush

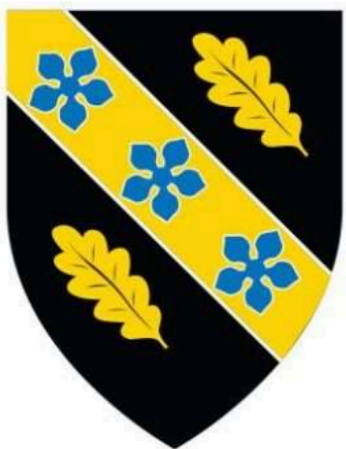
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LAB 4: Determination of Melting Point

L6 -Advanced Chemistry of OME (ACDA6020)

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Aim

Determine the melting point using Cole-Parmer M200 Melting Point Microscope (MPM) of four methyl-3-nitrobenzoate samples that were synthesised through nitration by four pairs of students at Cranfield University. Using the calculated melting point and the literature values, evaluate the purity of each sample provided.

Hypothesis

It is predicted that the four samples of methyl 3-nitrobenzoate collected will not have a sharp melting point but will exhibit a broad melting point that is lower than the expected value. There will be a slight variation between the samples, reflecting the differences in impurities present in each. Pure samples will have a narrow melting point range of 2°C within the literature value. [1] If the sample is impure, then the melting point is much broader at around 5°C from the literature value [2]. However, it is also predicted that at least one of these samples will have a melting point that is close to the literature value.

Variables

The independent variable is the ^{purity of} four samples that are being tested. The dependent variable is the melting points of each of the samples. The Controlled variables are the amount of each sample collected in the capillary tube, the calibration of the MPM, the heating rate of the MPM and the plateau temperature set.

Theory

The method of the experiment is shown in Appendix A

A simple definition of a melting point is when a substance changes from a solid phase to the liquid phase [3]. This happens when there is enough thermal energy acting on the substance to overcome the intermolecular forces between the particles, weakening the fixed, regular lattice structure of a solid and forming a disordered structure that allows the particles to move more freely, becoming a liquid [4]. A pure sample is a substance that only contains one compound or element [5]. In a pure solid, the intermolecular forces between the particles will all be equal, meaning the energy required to weaken the forces is also equal. When that amount of thermal energy is applied, all the intermolecular forces should be overcome at the same point, creating the sharp melting point as all the particles break from the lattice and become a liquid at the same temperature [6]

Impure solid samples contain a small number of other substances [7] within the solid lattice, making its lattice form less structured and more irregular, which causes weak points in the solid lattice. These weak points require less energy to break, which lowers the initial melting point of the sample. This also causes the lattice to have a range of different strength forces at different points, which widens the temperature range it takes to fully melt, broadening the melting point [8]. The extent of the broadening depends on the amount and type of impurities present.

The samples used in this experiment underwent recrystallisation before their melting point was tested. This is a purification technique used to help remove soluble impurities present in the solid. The sample is added to a hot solvent where it will be fully dissolved at a high heat then slowly cooled. As the solution cools, solid crystals form in the solvent while the impurities remain dissolved. Once the solution is fully cooled, it undergoes vacuum filtration, removing the solvent, leaving only crystals. These crystals are then dried, and the purity is determined by the melting point. [9] The slower the solution cools, the larger and more regular the crystals form [10]. This process only works if the impurity is soluble in a solvent or a mixture of solvents and often needs to be done multiple times before the sample is considered pure.

In OME (ordnance munitions and explosives) industry, recrystallisation is used to purify an energetic material to ensure long term stability, as impurities affect the material's sensitivity and lifespan, making them unpredictable and dangerous when impure. It is also used to control the crystals that are formed when it is required, as the size and shape of the crystal can affect the properties and stability of the energetic [12]

The melting point of pure Methyl 3-nitrobenzoate is between 78-80 °C [1][12]

Data

Sample	Appearance	Melting point, °C		
		Trial 1	Trial 2	Average
A	Pure White, clumpy and odorless	77	77	77.0
B	Pure White, not clumpy odorless	74	75	74.5
C	Yellow and clumpy gives off odor	74	73	73.5
D	Pure White, clumpy and odorless	77	77	77.0

Table 1 shows the results of the experiment:

duplicated tables!

Sample	Appearance	Melting point, °C		
		Trial 1	Trial 2	Average
A	Pure White, clumpy and odorless	77	77	77.0
B	Pure White, not clumpy odorless	74	75	74.5
C	Yellow and clumpy gives off odor	74	73	73.5
D	Pure White, clumpy and odorless	77	77	77.0

Table 1 - Sample appearance and melting point

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

Results and Discussion

The value for the theoretical melting point of pure Methyl-3-nitrobenzoate was 78-80 °C and is described as white/beige crystalline power [13]

Samples A and D had a melting point of 77°C, which is only 1-3°C under the expected value of 78-80°C. These are the closest to the literature value out of the 4 samples. There were still impurities within the samples, as they are under the expected value; however, these are minor. The appearance is as expected, so this and the melting point of these samples suggest that they are the purest samples collected.

Sample B, while showing the most promising physical characteristics of pure Methyl-3-nitrobenzoate, had a lower melting point of 74.5 °C. which is 3.5-5.5 °C lower than the literature value. This shows a larger number of impurities that caused a weaker lattice structure, lowering the melting point and slightly changing the sample's properties. This is a contaminated sample and would be classified as impure, though the impurity amounts are still low.

Sample C had a melting point of 73.5°C, which is 4.5-6.5°C under the literature value. It has no physical characteristics that match Methyl-3-nitrobenzoate. It can be concluded that this is a contaminated sample from this importation. The impurities present have affected the lattice structure significantly, which is seen in the

low melting point. The discolouration also suggests there are unreacted materials present in the sample

Potential sources of ^{error} ~~effort~~ in this experiment include a variation in the patching of the capillary tube, minor differences in amounts, ^{but minimal} packing density and location in the tube could have an impact on the heating rate of the material. There is also a source of observational error, as the experiment relies on the operator to identify the first signs of melting. There is also a possibility of residual solvent left if the drying process was not fully complete, which could lower the melting point. This is likely the reason for the lower rate in samples A and D.

Overall, the result supports the hypothesis that the samples will have a lower melting point than the expected value. There was also the predicted variation among the samples; however, there were two samples that could be considered pure, which is an improvement on the original hypothesis. This suggests that the synthesis of Methyl-3-nitrobenzoate was a success.

Conclusion

The results show that samples A and D were the purest samples as they were only 1°C away from the expected value; the other samples contained high levels of impurities. These results support the original hypothesis that most samples would show broader and lower melting points due to contamination. Overall, melting-point determination proved to be an effective method for assessing sample purity and evaluating the success of the synthesis.

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access dates are missing for all online sources

Appendix A

Lab tools

Cole-Parmer M200 Melting Point Microscope (MPM)

Glass capillaries

Glass dish

Four samples of methyl-3-nitrobenzoate

Operation

1. Set up the MPM by connecting to the power supplier and turning it on. The display should show a temperate and the sample chamber light should be on
2. Weigh the sample and take notes on the appearance of the sample, and if it is in line with what is expected
3. Place the sample on a glass dish and lightly tap the sample with the open end of the capillary tube to collect the sample. When the sample is collected, tap the capillary tube on the closed end to bring the sample to the middle of the tube. Take two trial capillary tubes for each sample, so there should be a total of 8 tests
4. Set the plateau temperature of the device by holding the plateau button and then adjusting using the arrow keys. Release the button when the temperature is set at 10°C below the expected melting point, in this case 68 °C.
5. Place the capillary tube in the sample hole and check that it is visible through the lens. The test is ready to begin, and the start button can be pressed on the machine. It should quickly heat to the plateau temperature set, and a light should turn on to signal that it is at the temperature.
6. Press starts again to initiate slow heating of 2°C/min, then the operator must look through the lens and record the temperature the sample is at when the first sign of melting is visible
7. Press stops after the observation is collected and allow the machine to cool before removing the sample.
8. Repeat steps for all 8 of the trials before allowing the machine to cool and switching off

FINAL GRADE

GENERAL COMMENTS

61 /100

Engagement with Literature 8/15

Relevant literature values for the melting point of methyl-3-nitrobenzoate are correctly reported and supported. Sources are generally credible; however, the reference list relies heavily on websites, with very limited use of peer-reviewed journal literature. Literature is mainly used descriptively in the introduction rather than to interpret or explain the experimental results. Several online sources are missing access dates, which weakens formal IEEE compliance.

Knowledge and Understanding 15/20

The aim of the experiment is clear and appropriate. Melting point is correctly defined and well explained in terms of crystal structure and intermolecular forces, and the effects of impurities on melting point depression and range broadening are accurately described. Recrystallisation is correctly understood as a purification method, with clear explanation of why impurities remain in the solvent and how this influences melting behaviour. Greater precision in defining the independent variable (purity rather than sample identity) would strengthen this section further.

Cognitive and Intellectual Skills 11/20

A hypothesis is stated but lacks clarity and specificity regarding expected melting behaviour. Results are linked back to the hypothesis, and experimental uncertainty and subjectivity in visual melting point determination are acknowledged. However, the discussion remains largely descriptive, with limited evaluation of the significance of deviations from literature values or consideration of melting onset versus completion.

Practical Application Skills 17/30

The experimental procedure is clearly described and appropriately placed in the appendix. Duplicate measurements were performed with reasonable agreement. However, only melting point midpoints are reported rather than full melting ranges, limiting the quality of purity assessment. The relevance of melting point testing to energetic materials is implied but not

developed in detail, and no complementary analytical methods are discussed.

Transferable Skills 10/15

The report is clearly structured and written in a professional tone. Tables are properly titled, although a duplicate table indicates a lack of final proofreading. The word count is slightly above the agreed limit.

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