

MF LAB 4 report

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ACDA6020 LAB #4

Melting point of Methyl 3 Nitrobenzoate

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Introduction

Samples of Methyl 3 Nitrobenzoate (M3NB) were prepared by students at Cranfield university's teaching laboratory and returned to UWTSD for assessment. These samples were then assessed for their appearance, crystal structure and melting point. Melting Point determination is commonly used for assessment of the purity of organic materials as impurities can lower melting point significantly. This is relevant to OME as purity has a significant effect on the stability and performance of energetic materials throughout their life.

Background

Samples M3NB ^{were} prepared during a visit to the Cranfield university's teaching lab at the Defence Academy of the United Kingdom (Shrivenham). M3NB was prepared by nitrating methyl benzoate using a mixture of nitric and sulphuric acid. M3NB is not an energetic substance itself but is analogous to Nitrotoluene (methyl -3-nitrobenzene), a precursor to energetics such as Trinitrotoluene (TNT) and Dinitrotoluene (DNT) which are used as an explosive and as energetic propellant additives respectively. Figure 1 and Figure 2 shows the similarities between the two chemical structures.

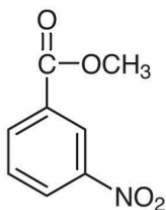


Figure 1 Methyl-3-Nitrobenzoate [9]

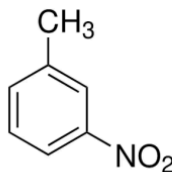


Figure 2 3-Nitrotoluene [8]

Pure M3NB forms a white crystalline power [1], whilst Methyl dinitrobenzoate forms a white to off – white powder [2], Methyl Trinitrobenzoate is possible but is relatively rare in literature. ^(Ref) The melting point of M3NB is expected to be 78-80°C [1] whilst the melting point of methyl dinitrobenzoate is between 107-109°C [3].

Purity is essential to OME as it directly corresponds to the sensitivity of materials; higher purity leads to more predictable behaviour [4] and reduced formation of hotspots or other undesirable phenomena.

The melting point of a solid is the temperature at which its structure breaks down and transitions to its liquid phase. In pure organic compounds crystal structures are highly ordered and require significant energy to overcome intermolecular forces; this is highly repeatable allowing high confidence when comparing to literature values. Impurities disrupt this structure, reducing the strength of intermolecular forces, in turn reducing the melting temperature[5] and allowing this to be used as a measure of purity. Impurities also increase the spread of melting points; creating a range instead of a precise point; this can also be an indicator of contamination.

Variables:

Independent variable:

- Identity of the sample (A, B, C, D).

Dependent variable:

- Melting point (°C).

Controlled variables:

- Heating rate,
- plateau temperature,
- sample quantity,
- instrument settings

Hypothesis

It is expected that if the samples are pure, they will present as small white crystals, with a melting point consistent with reported literature values: between 78-80°C. If methyl dinitrobenzoate is present, whilst having a higher melting point the overall melting point of the resulting mixture will likely have a lower melting point as the regular crystal packing would be disrupted. Reducing the energy needed to break up the crystal's intermolecular forces [6] Impurities increase disorder in the solid phase, reducing lattice stability and lowering the Gibbs free energy difference between solid and liquid. [6] *what is Gibbs free energy and why is it mentioned here?*

Results

Four samples were analysed with each measured twice to obtain the following results:

Table 1 Melting Point results

Sample	appearance	Trial1 (mp, °C)	Trial 2 (mp, °C)	Average (mp, °C)
A	White fluffy powder with short and stout crystals	77	76	76.5
B	White fluffy powder with short defined crystals	77	77	77
C	Cream fluffy powder, somewhat clumpy wider crystals than sample 1.	75	75	75
D	Yellow clumps, short and very wide crystals	72	71	71.5

The following figures show the morphology of the crystal samples.

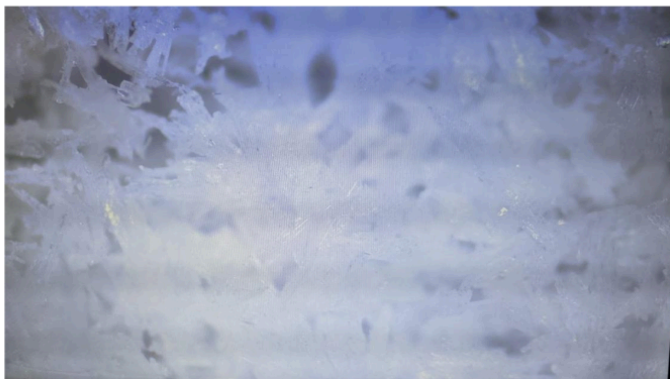


Figure 3 Sample A crystals



Figure 4 Sample B crystals

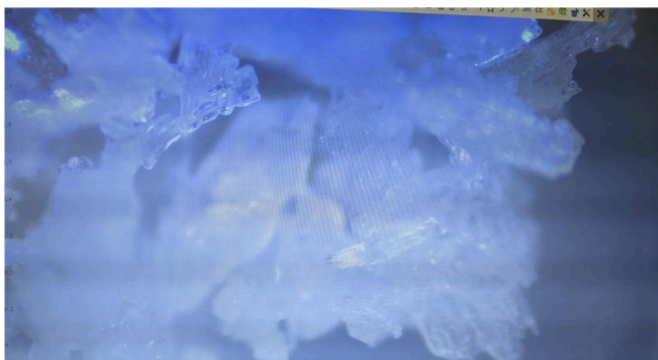


Figure 5 Sample C crystals



Figure 6 Sample D crystals

Samples A and B appeared bright white with well-defined crystals. Sample C was off-white with shorter, wider crystals, while sample D was distinctly yellow and formed large clumps. These visual differences corresponded closely with the melting point trends.

Discussion

All samples exhibited melting points below the literature range for pure M3NB (78–80°C). Samples A&B were closest to the expected range with the 76.5°C and 77°C respectively. Both samples were bright white, and whilst the crystal morphology is minorly different, they shared many of the same properties. This indicates a comparatively high degree of sample purity with only trace impurities.

Sample C, produced a lower melting point as well as being notably discoloured, appearing cream/off-white. This likely due to partial over-nitration causing the appearance of methyl dinitrobenzoate in the substance. Despite having a higher melting point, methyl dinitrobenzoate would lower the melting point of the overall substance due to its disruption of the crystal packing of M3NB.

Sample D displayed the lowest melting point and a pronounced yellow colour. This suggests significant contamination from methyl dinitrobenzoate, residual solvents and unreacted methyl benzoate. The clumping behaviour reinforces this as methyl benzoate's oily [7] properties may cause it to act as a binder, further reducing lattice stability and the substance melting point.

The small number of data points for each sample limits the confidence, but this could be improved through additional repetitions or the use of methods such as FTIR to identify impurities. This means that the purity cannot be reliably assessed by looking at the range of melting points.

Conclusion

In conclusion, the above results show the melting point of a range of methyl-3- nitrobenzoate samples. There is a range of purities with several samples containing indications of contaminants such as the products of over nitration or residual reactants and solvents. Overall, two samples likely contain M3NB of a high purity (A&B). But further testing would be recommended to identify the cause of difference from literature values. The differences in the crystal morphology and visible properties of the materials are also highlighted in the differences in melting point; it provides an important reminder that manufacture of high purity chemicals is a difficult task, this is especially important in OME where production of energetics rely on high quality materials to maintain performance, and safety requirements.

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Appendix:

A1: Method

PPE: Gloves, lab coats, safety goggles, (long hair tied back).

A small quantity of M3NB produced by UWTSD students during the visit to the Cranfield university teaching laboratory, was placed onto a sample plate. 4 samples were collected for this analysis.

A small quantity was added to a hollow glass capillary tube. Only a few crystals are required for each test.

For this experiment a Cole-Parmer M200 Melting point microscope (MPM) was used.

The MPM was preheated to 60°C.

The sample was inserted into one of two identical slots and the MPM set to ramping mode. This would slowly increase the temperature until the sample under observation melted.

Each sample was tested twice and an average taken for the melting point.

Additionally, each of the 4 samples was observed via an optical microscope. The make and model were not recorded.

FINAL GRADE

71 /100

GENERAL COMMENTS

Engagement with Literature 9/15

The literature melting point for methyl-3-nitrobenzoate is correctly reported and supported by a reference. A range of sources is used; however, these are predominantly online resources, with relatively few credible books and peer-reviewed journal articles. A reference supporting statements about the abundance or formation of methyl trinitrobenzoate is missing. Literature is largely listed rather than used to interpret experimental results and is mainly confined to the theory section, with minimal use in the discussion. There are also inconsistencies and formatting errors in the application of IEEE referencing.

Knowledge and Understanding 14/20

You correctly recognise that impurities decrease the melting point and provide clear definitions of melting point and melting range. The report demonstrates a good understanding of why pure compounds melt sharply and gives correct explanations of melting point depression and melting range broadening. The role of intermolecular forces is appropriately mentioned, and variables are correctly identified.

However, Gibbs free energy is referenced without explanation or application, which limits the depth of theoretical understanding demonstrated at L6.

Cognitive and Intellectual Skills 15/20

A detailed hypothesis is clearly stated. Experimental values are compared with literature data, and differences between samples are analysed logically. There is a good attempt to explain clumping behaviour, and experimental uncertainty is acknowledged due to the small number of data points. The proposal of alternative analytical methods demonstrates reflective thinking.

However, the discussion focuses on melting point midpoints rather than full melting ranges, which limits the strength of the purity assessment.

Practical Application Skills 21/30

The methodology is provided in the appendix. The importance of purity in OME materials is identified in the introduction and developed further in the background section, with clear links made to predictable behaviour and reduced hotspot formation. Raw data are presented using correct units and significant figures.

More explicit discussion of heating rate control and instrument limitations would further strengthen the demonstration of practical competence.

Transferable Skills 12/15

The report meets the word count requirements and follows the required structure, with a clear separation between Results and Discussion and a logical progression between sections. Tables are clearly labelled with units, figures are appropriately captioned, and formatting is consistent throughout. There is, however, some unnecessary narrative in the introduction and background sections, which could be reduced to improve conciseness.

PAGE 1

PAGE 2

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