

Lab 4

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Our Reference: ACLAB_03

LAB REPORT ON THE DETERMINATION OF BOILING POINT FOR METHYL-3-NITROBENZOATE

References. See footnotes and embedded documents section at end of document.

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the content of the Appendix shows the experimental setup and procedure for mp determination, not preparation of M3NB

1. Introduction and Background

The melting point of a substance is a basic but clear way of demonstrating a compound's purity. This is because a narrow range of a precise melting point is indicative of a pure sample. The lower the melting point and further away it is from ideal is largely down to the contamination of the compound. [1]

There are many different styles of experiment to measure the melting point of a substance but they all rely on the basic principle of observing the change from a solid to a liquid as the temperature increases. This change is shown below.

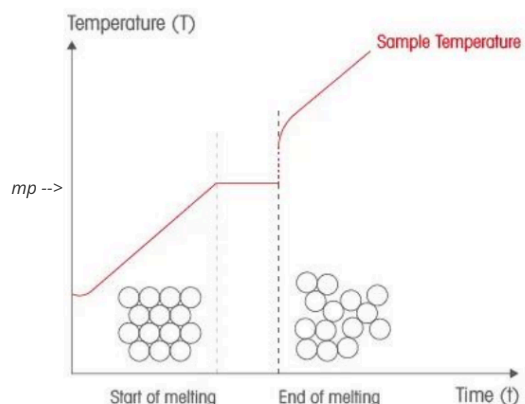


Figure 1 - Temperature Time graph of a melting substance [2]

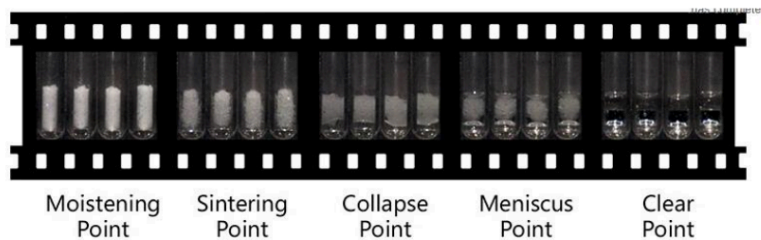


Figure 2 - Melting Point Stages [2]

1.1. Melting Point in OME

The determination of melting point of an energetic material is one piece of critical information in understanding the initiation criteria, performance and processing abilities. The melting point is generally not determined separately from other information but instead is often part of a bigger test. The most common test conducted that archives the melting point is Differential Scanning Calorimetry (DSC)

DSC is a method of small scale testing that measures the heat flow in to or out of a material as it is heated or cooled. This measurement can reveal key events such as decomposition, melting or glass transition. [3] [4]

Although the capillary method used in this experiment is basic the same principle can be further improved using an adjustable microscope. Slightly more advanced method is referred to as Hot Stage Microscopy (HSM). HSM is widely used to observe a variety of thermal transitions in energetic materials as it enables the observation of phenomena such as foaming and sintering. [5]

2. Purpose and Aim

The intent of this experiment was to determine the melting point and therefore indicate purity of the Methyl-3-Nitrobenzoate synthesized by the student on 19th November 2025.

The hypothesis is that a value in the range of 71-78°C will be achieved for the melting point as the substance is likely to contain some impurities. *why this range is so wide? [Ref]*

3. Setup and Procedure

The full procedure can be found at Annex 1

For this experiment the initial moistening / sintering stage was observed and recorded as the melting point as this stage is the first point that is easily observable in the equipment available.

For Methyl Benzoate (MB) the melting point is -12.3°C. [6]

For Methyl-3-Nitrobenzoate (MNB) the melting point is 78°C. [7]

As the likely main contaminant in MNB is MB this is why there would be a reduction in melting temperature if there is the presence of MB in the sample.

As the samples were expected to be impure a plateau temperature of 70°C was used. From here the ramp function was initiated until melting was observed.

In addition to determining the melting point the samples were placed under a microscope to understand the visual appearance, crystal structure and possible impurities.

4. Visual Results**4.1.CH/OM**

The sample below shows a slightly yellow crystalline structure with a fine and even crystal size.

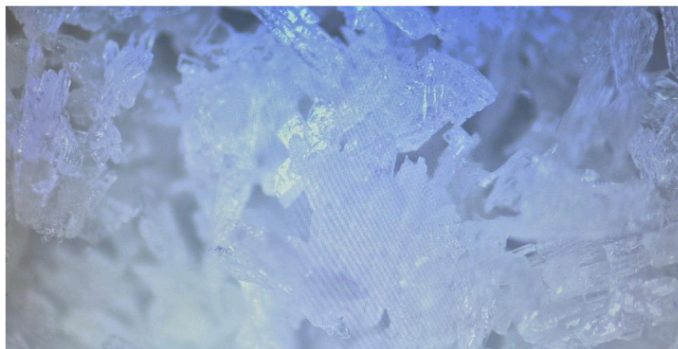


Figure 3 - CH/OM Sample

4.2.LB/DG

The sample below shows a slightly yellow structure with even finer crystals of an even size.

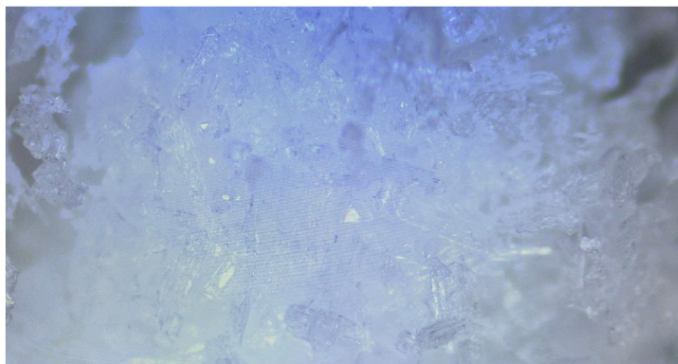


Figure 4 - LB/DG Sample

4.3.JE/RD

This sample demonstrated the whitest crystals that were the smallest and very even giving confidence that this was the purest sample.



Figure 5 - JE/RD Sample

4.4.LM/NC

This sample showed a significant yellow/ brown hue with significant clumping and large crystals. There was significant variation in crystal size and the sample exuded an odour. This provides confidence that this sample was the least pure.

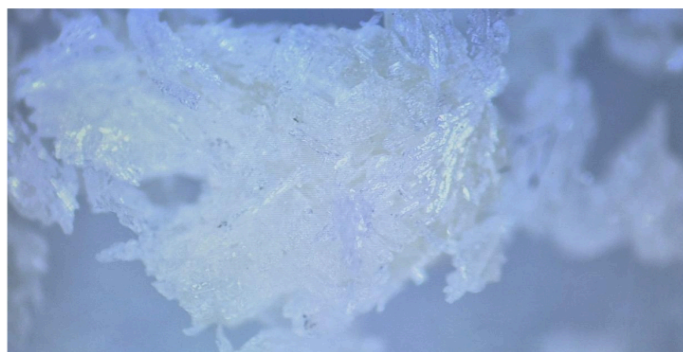


Figure 6 - LM/NC Sample

5. Results

The summarised visual appearances and melting point data are shown in Table 1 for the 4 samples. Comparing and contrasting the results the samples can be ordered in terms of purity below.

Sample	Rank	Visual Appearance	MP (°C)		MP (°C)	Δ from Ideal (°C)
			Trial 1	Trial 2		
JE/RD	1	White Fluffy Crystals, Minimal clumping	77	77	77	1.0
LB/DG	2	Yellow Hue, Very Fine Crystals & Compacted	76	77	76.5	1.5
CH/OM	3	Slightly Yellow, Fine Crystals & Fluffy	75	75	75	3.0
LM/NC	4	Yellow, Clumped & Odorous	72	72	72	6.0

Table 1 - Results Summary
you should have recorded the range of temperatures from the first signs of melting till all sample turned into liquid

The results shown in Table 1 demonstrate a varying level of quality across all the samples with a top ranked sample coming within 1°C of the ideal value. On the other end of the spectrum the least pure sample melting point is significantly different from the ideal value with a delta of 6°C. this is additionally supported by the change in appearance with the sample containing a brown hue indicating un nitrated compound.

Within this experiment the key sources of error are largely human based, as the determination of melting point is based upon a visual change when an individual determines that change can differ between individuals. To best limit this error the same observer was used each time. The next largest form of error was the quantity of material placed within the capillary tube.

6. Conclusions

The results achieved support the hypothesis in that all results were within the expected range of -10% of the ideal melting point 78°C. In general the samples where all an acceptable purity excluding the LM/NC sample. This sample had a clear indication of un nitrated base material remaining within the sample. This is what gave it the brown hue, odour and low melting point. The top-ranking sample achieved a near pure sample with a melting point of 77°C. This is indicative of a full nitration and effective recrystallization stages.

To further improve the results of this experiment additional measurements could be taken across the 4 samples. 2 measurements is not a statistically relevant sample as an average of 2 is rather meaningless. In automated melting point systems as many as 6 samples can be tested simultaneously for faster more reliable results. [8]

Lastly if the true aim of measuring the melting point is to determine purity other methods such as FTIR analysis could be used to fully understand the level of contamination and what compounds are contaminating the sample. This provide more information over the 'is the sample contaminated or not'.

7. References

- [1] Stanford Research Systems, "Melting Point Determination," Lambda Photometrics Limited, *Harpenderis this an online source?*
- [2] Mettler Toledo, "What is Melting Point?," Mettler Toledo, [Online]. Available: https://www.mt.com/int/ar/home/applications/Application_Browse_Laboratory_Analytics/Thermal_Values/melting-point-determination.html. [Accessed 27 11 2025].
- [3] C.-j. Zuo and C.-y. Zhang, "Standardizing differential scanning calorimetry (DSC) thermal," *Energetic Materials Frontiers*, vol. 6, no. 2, pp. 189-194, 2025.
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- [10] M. WARMAN, S. SEMEL, J. LECCACORVI, R. MOTTO and R. HUTCHINSON, "CONVERSION OF GUANIDINE NITRATE TO NITROGUANIDINE WITH RECYCLE OF SPENT ACID," Technical Library, 1975 *no need for all CAPITALS*
- [11] P. Worsfold, A. Townshend and C. Poole, *Encyclopedia of Analytical Science*, Elsevier Ltd, 2005.

sources 9-11 not mentioned in the text

8. Annex 1 - Methyl-3-Nitrobenzoate Preparation Procedure

University of Wales Trinity Saint David – OME Programme

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.

3

Figure 7 – Melting Point Procedure

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REPEAT THE MEASUREMENTS TWO TIMES FOR EACH SAMPLE.

Literature values of melting points:

	melting point, °C
methyl benzoate	-12°C
methyl-3-nitrobenzoate	78-80°C

Experimental values of melting points 76°-79° or 78-79.

Sample	appearance	trial 1	trial 2
		mp, °C	mp, °C
A CH ₃ OH	11.5036 - no cap 12.3212 - cap	75	75
B LB/DG		76	77
C JE/RD		77	77
D LM/KC		72	72

OBSERVATIONS AND OTHER RELEVANT INFORMATION plateau = 2°C

Description / notes

A - slightly yellow fine crystals

B - ~~slightly~~ slightly yellow fine crystals

C - white slightly crystals minimal cap

D - yellow, clumped, porous, odour

4

Figure 8 – Melting Point Raw Results

53/100

Engagement with Literature 7/15

Relevant literature values for the melting points of methyl benzoate and methyl-3-nitrobenzoate are reported and supported by references. There is evidence of independent reading, including mention of techniques such as DSC and HSM. However, the depth of engagement with the literature is limited. Most sources are online databases or company material, with very few peer-reviewed journal articles. Literature is mainly presented in the introduction and is not consistently used to interpret or strengthen the discussion of experimental results. Several references listed are not cited in the text, and there are inconsistencies in IEEE formatting, including missing access dates and incomplete reference details.

Knowledge and Understanding 9/20

The aim of the experiment is clearly stated and a definition of melting point is provided. A basic connection between melting point and purity is made, and the presence of melting point depression due to impurities is recognised. However, this relationship is not explained in sufficient depth. Key concepts such as crystal lattice disruption and intermolecular forces are not discussed, limiting the scientific explanation of the observed trends. The melting range for a pure compound is inaccurately described as broad, and recrystallisation is only implicitly linked to purification rather than explicitly explained. Overall understanding is sound at a descriptive level but lacks conceptual depth.

Cognitive and Intellectual Skills 12/20

The hypothesis is stated, and experimental melting point values are compared with literature data. Samples are logically ranked by inferred purity, and differences in appearance are appropriately linked to melting behaviour. Claims are generally supported by the data presented. However, the hypothesis does not clearly justify the expected temperature range, and discussion focuses on melting point midpoints rather than full melting ranges. Evaluation of uncertainty is limited, with emphasis mainly on human observational error, while other factors such as sample quantity and packing effects are not considered. The discussion would benefit from deeper evaluation of the reliability of the conclusions.

Practical Application Skills 15/30

The relevance of melting point testing to the safety and stability of energetic materials is mentioned, but this link is not developed in depth. Experimental measurements were repeated, with excellent agreement between trials, and sample appearances are clearly described. However, the experimental procedure is included as a copied screenshot rather than a clearly written methodology section, which reduces professionalism. Instrumental limitations and heating-rate effects are not explicitly discussed, and no alternative analytical techniques are proposed to support or validate the results.

Transferable Skills 10/15

The report follows the required overall structure and remains within the agreed word count. Tables and figures are clearly labelled and appropriately referenced. However, the procedure section is not placed in an appendix, and some wording is overly informal for a scientific report (e.g. "rather meaningless"). While the report is generally readable, clarity and professionalism could be improved through more precise language and tighter structuring.

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