

ACDA6020 LAB 4

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Figure 1 Cole Parmer M200 MPM [8]

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DETERMINATION OF MELTING POINT

Lab session 4

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1. Introduction

This lab report details the use of the Melting Point Microscope (MPM) to determine the melting point of 4 samples of a known compound.

1.1 Aim

To determine the melting point of 4 samples of methyl-3-nitrobenzoate, each sample synthesised by separately to evaluate the purity of each sample.

1.2 Hypothesis

Based on the appearance of the samples it is expected that samples A and B are expected to be of high purity due to their bright white fluffy appearance, while samples C and D are expected to be of lower quality due to their yellowy and clumpy appearance with D also having an additional strong odor. It is the hypotheses that samples A and B are the purest samples with a melting point close to the literary value while sample D is the least pure, or most contaminated with a melting point much lower than the literary value.

1.3 Variables

The variables if this experiment consisted of:

- Independent:** Sample. This includes the natural variation between humans. Each sample was synthesized by a different group or person. It is this variation and adherence to the procedure or lack of, where the contaminants are introduced.
- Dependent:** Temperature of state change, from solid to liquid. Although this could be debated due to again the natural human error of how quickly or consistently the state change is recorded.
- Control:** Sample size and temperature range over time. The MPM sample tubes and the MPM correct set up mechanically and physically control both factors.

2. Theory

M3NB (which is not explosive)

The morphology of the ~~explosives~~ examined during this experiment along with the melting point can give an indication of impurities, although to reiterate only an indication, this is due to the many crystallization methods for explosives [1] and it has been shown that mean particle size can also have an effect on both crystalline structure and melting point [2], this can be seen well within RDX when the crystalline structure is a spherical hexagon RDX/TNT vs standard RDX/TNT there are less crystal defects and a melting point increase of 1.57°C [3] [4].

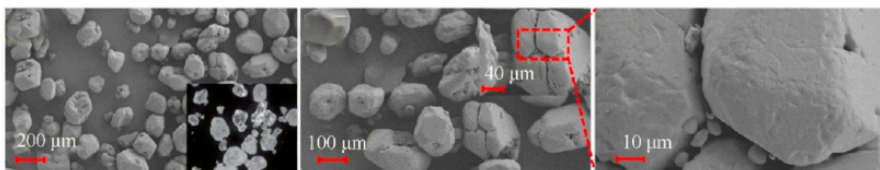


Figure 2 Raw RDX Crystals. Uneven particle & size distribution of about 50–240 μm [9]

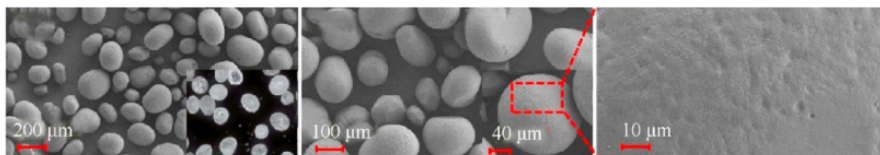


Figure 3 Spherical hexagon RDX using RAM. Uniformed particle & size distribution of 100–260 μm [9]

Figures 2 and 3 show raw and spherical RDX crystals under scanning electron microscope. Although the crystal structure has been changed using RAM and a solvent the theory of the crystal structure remains the same with a reduction of crystal defects as previously mentioned. Typical phase change of a pure solid to liquid is a sharp point well documented within literature for that particular solid. When impurities are introduced, this reduces the melting point, typically the more impurities there are, the broader the melting point range known as melting point depression [5].

During the recrystallisation process when synthesizing explosives, commonly the solvents used can be left within the explosive and disrupt the uniformity of the crystal structure. In the regions of impurities, the weaker intermolecular interactions require less thermal energy to break apart, causing the reduction of melting point. These impurities impact mechanical sensitivity, through the formation of hotspots [1]. Therefore, highlighting the importance of testing explosive purity.

3. Materials & Method

The materials and method required for this task can be found in Appendix 1.

4. Observations & Data

Sample:	Appearance and crystal structure.	Trial 1	Trial 2
		Melting Point °C	
A	White powder, fluffy short defined crystals. Fat needles	77	76
B	White fluffy powder, short defined crystals.	77	77
C	Cream solid clumps, crystals much fatter than A&B	75	75
D	Yellowy large clumps, very wide crystals. Strong Odor	72	71

Table 1 Experimental values of melting points
 you should have recorded the range of temperatures from the first signs of melting till all sample turned into liquid
 Samples A and B show melting points within 1°C of the literature values. While sample C shows a result 3°C lower and sample D, 7°C lower.

Substance	Melting point °C
Methyl benzoate	-12 [6]
Methyl-3-nitrobenzoate	78-80 [7]

Table 2 Melting Point: literary values

6. Discussion

As seen in table 1, samples A and B are within 1°C of the literature values seen in table 2, this demonstrates samples A and B are pure samples and could be used within an explosive with a high degree of confidence of not impacting safety. Samples C and D show -3°C and -7°C from the melting point, if being used for explosives a recommendation of either recrystallisation or disposal would be made. As mentioned in section 2, the melting point depression of these two samples indicates a large deviation from the sharp melting point demonstrating a high level of impurities, if used within an explosive these impurities would induce hotspots and negatively impact mechanical sensitivity.

7. Conclusion

Overall, the experiment demonstrated that melting point analysis is an effective method for assessing the purity of synthesised compounds. The results highlight the importance of controlled synthesis and recrystallisation, especially poignant for energetic materials where impurities negatively affect mechanical sensitivity and safety.

The observed link between morphology, appearance and melting point is supported by literature, impurities disrupt crystal uniformity and weaken intermolecular bonds resulting in lower melting points. Sample D showed irregular morphology, large fat crystals and a strong odour was confirmed to be the least pure sample, consistent with the hypothesis.

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Appendix 1.

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.

Figure 2 Materials & Method

FINAL GRADE

62/100

GENERAL COMMENTS

Engagement with Literature 10/15

The literature melting points for methyl-3-nitrobenzoate and methyl benzoate are correctly reported and supported by appropriate sources. A range of credible references is used, and citation style is generally consistent. However, the literature is used mainly to provide background context in the introduction and theory sections. There is limited use of literature to interpret or explain the experimental results in the discussion. Greater critical engagement with sources when analysing the data would strengthen this section.

Knowledge and Understanding 14/20

The aim of the experiment is clear and appropriately defined. Key variables are identified, and melting point is correctly defined and linked to sample purity. Melting point depression is explained in terms of disruption to crystal structure, and intermolecular forces are used to justify differences between pure and impure samples. Recrystallisation is correctly recognised as a purification method. Further depth in the discussion of intermolecular forces and their relative strengths would improve this section.

Cognitive and Intellectual Skills 11/20

A clear hypothesis is stated based on the visual appearance of the samples, and results are consistently linked back to this hypothesis. Samples are ranked by inferred purity, and conclusions are generally supported by the experimental data. However, the analysis remains relatively surface-level. Melting point midpoints are discussed rather than full melting ranges, and limitations of the method are not critically evaluated. Discussion of SEM and RAM is not directly relevant to this experiment and does not add value to the interpretation.

Practical Application Skills 17/30

The experimental procedure is included in the appendix; however, copying it as a screenshot reduces

professionalism. The impact of impurities on energetic materials is explained, and real-world relevance to explosives is demonstrated. Duplicate measurements show good agreement, and samples are ranked logically by inferred purity. The discussion would be strengthened by a clearer evaluation of experimental limitations and by suggesting complementary analytical techniques such as DSC or FT-IR.

Transferable Skills 10/15

The report is below the agreed word count, which limits the depth of analysis. Tables and figures are clearly captioned, and a formal scientific tone is generally maintained. However, clarity of writing is inconsistent in places, and some sentences would benefit from improved structure and precision.

PAGE 1

PAGE 2

PAGE 3

Text Comment. ???

Text Comment. M3NB (which s not explosive)

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Text Comment. these are beautiful SEM images, but pictures of sampels A-D would be more reevant in this report

PAGE 4

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

PAGE 5

PAGE 6
