Name__________________

Melting Point Study of A Solventless Reaction

(adapted from Doxsee, K.M. and Hutchison, J.E., *Green Organic Chemistry*)

Techniques

Interpreting a Handbook (Chapter 3)
The Melting Point Experiment (Chapter 12– Mel-Temp apparatus)
Recrystallization (Chapter 13 pp 109 – 112)

Green Principles

Atom Economy
Solventless Reactions
Catalysis

Introduction

Melting point (m.p.) evaluation is a simple, yet powerful tool to evaluate chemical reactions.

All crystalline solids have a specific melting point or range that is determined by molecular structure and crystal packing. A melting point is the temperature at which a pure crystalline solid melts. Sometimes only a melting range can be determined. A melting range starts at the temperature the first crystal starts to melt and ends at the temperature the last crystal melts. For some compounds only a melting range can be determined. If this is a pure compound the melting range will be small (2 – 3 °C).

Melting points are not unique, but when the possible compounds are known, the melting points are usually distinctly different and can be used to identify the solid. By comparing your measured melting point to the reference value, you can confirm that you have the compound you expected. If the value is low and/or the melting range covers several °C, the compound is probably impure. This is an example of colligative properties which you learned about in general chemistry. Melting point depression is dependent upon the number of solute particles in the solvent. In general, the more impure the solid is the lower the melting point and the broader the range. On the other hand, if you have a melting point that is different from the expected value and not a melting range, then the product is something other than what you predicted.

Discussion

The reaction you will be doing for this experiment is an Aldol Condensation reaction. Aldol reactions are a powerful tool for forming carbon-carbon bonds and are used in many important synthesis processes. The formation of carbon-carbon bonds is very important to the success of synthetic organic chemistry. For the purpose of this experiment, we are not concerned about the details of this reaction. Our focus is on using melting points as a way to evaluate a chemical reaction. Don't worry, we will revisit Aldol condensation reactions later in the year and study them in more detail. The reaction you are

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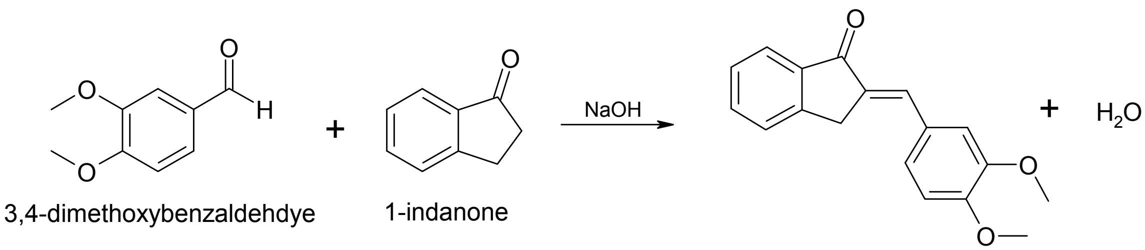
doing today looks impressive (and is) as you bond two complex molecules to form a larger, more complex product. The reaction is:



Note that the reactants are given common names rather than IUPAC names. Also, the product is complex enough that it is not named here. Many compounds with benzene rings in the structure are referred to by their common names rather than the IUPAC name as the common name is simpler.

In this reaction you will be crushing the two solid reactants together. As they mix they will have their melting points will lower (colligative properties) and at some point they will become a gummy liquid or oil. As you continue to mix the reactants, now with a catalyst added, the reaction will continue to take place and a new solid will form. Once the reaction is complete there will only be solid present. You will then be able to evaluate the purity of that solid based off the melting range.

Greening the Chemistry

This experiment demonstrates three green principles. First this reaction demonstrates excellent atom economy. Atom economy is a comparison evaluating how many atoms in the reactants end up in the product. In our reaction, all the atoms in the reactants go into the desired product except for two hydrogen and one oxygen atoms. This is an extremely efficient reaction, or as we say, has high atom economy. The atoms not going into the desired product form water – not bad as waste goes.

Secondly this reaction is also carried out without a solvent. As you will see throughout this year, most reactions use solvents. Solvents are a significant component of industrial chemical waste. Making a reaction solventless significantly reduces chemical waste.

Thirdly this reaction uses sodium hydroxide as a catalyst. A catalyst increases the rate of reaction, without being consumed by the reaction, allowing a small amount of catalyst to be recycled many times within a reaction. The catalyst does this by creating a different reaction pathway with a lower energy of activation barrier and/or less steps making the reaction faster. The catalyst does react during this process but is regenerated before the product is formed allowing it to react again and again.

Solventless Reactions: A Melting Point Study

Experimental

SAFETY PRECAUTIONS: Avoid contact with the NaOH as it is caustic. Wear gloves when doing this experiment.

Take a small sample of 3,4-dimethoxybenzaldehyde and a small sample of 1-indanone and determine their melting points. Using weighing paper, weigh 0.25 g of 3,4-dimethoxybenzaldehyde and 0.20 g of 1-indanone and place them in a small beaker. Crush and scrape the two solids together with a metal spatula until they become a darkly colored oil. Be patient as the mixing will take some time. Note that pressing harder will not speed the process, but may break the beaker. Once the oil has formed add 0.05 g of finely ground solid NaOH (prepared using a mortar and pestle). Continue mixing and scraping until the mixture becomes solid. Let the mixture stand for 15 minutes to allow the reaction to come to a stop.

Add about 2 mL of 10% aqueous HCl solution. Scrape the beaker thoroughly to make sure that all the solid is mixed with the solution. Check the pH of the solution by dipping your spatula in the solution and then touching it to the pH paper. Confirm that the solution is acidic, if not add more acid and mix again. Collect the product using vacuum filtration. Draw air through the product for a few minutes to help dry the product and then dry the product for an additional 15 minutes in the drying oven.

Determine the mass and melting range of your product. Pure product will have a melting range of 178 – 181 °C.

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Experiment Report

For this experiment I would like you to create a summary report. The report should be a typed one-page narrative and should include:

- A brief summary of the experiment (one paragraph)
- A discussion of your results that should include:
  - Melting points
  - Amount of product collected and its melting range
  - Percent yield of product from your starting materials
- An analysis of error and the quality of your experiment. Did the experiment go well or poorly and can you explain what went wrong? Do you find your results to be reliable?
- An evaluation of this experiment in terms of its greenness.

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Pre-Laboratory Questions

1. Briefly describe how you load a melting point tube.

2. Briefly describe how you efficiently determine a melting point in terms of temperature rise on the Mel-temp apparatus.

3. Complete the following table for chemical data

Compound	**Molecular Wt.**	**m.p. °C**	**Water Soluble?**
3,4-dimethoxybenzaldehyde			
1-indanone			
NaOH			
Product			

4. Sodium hydroxide is both caustic and hygroscopic. Define these terms.

Caustic:

Hygroscopic:

5. What safety precautions should you take during this experiment?

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