

NOTES ON EXPERIMENT 3

Nitration of Methyl Benzoate

Take great care when working with concentrated sulfuric or nitric acids. These acids can burn your skin badly, and a splash in your eye could blind you permanently- so wear your safety glasses or goggles, and gloves. The acids also react violently if water is added to them- always add the acid into water, not the other way round. Make sure that you make up the 0.2ml sulfuric acid/0.2ml nitric acid in a **separate** tube. It is best if they react to form the nitronium ion before being added to the methyl benzoate.

As with all reactions where solids and liquids are present in a reaction mixture, it is important to mix well to avoid impurity formation and incomplete reactions.

After the nitric acid has been added, you may save time by warming the mixture to room temperature with a beaker of room temperature water. However you must time the 15 minutes reaction time from when it reached room temperature, not before. I would in fact recommend a reaction time of 20-25 minutes at room temperature. While this may add 5-10 minutes, you should realize that an incomplete reaction (which students often have, unfortunately) causes the product to oil out as a solution in methyl benzoate. For example even a 50% complete reaction will lead to no isolated product at all- remember methyl benzoate is a liquid- and you will have to start over.

Wash well with water, otherwise residual acid will decompose your product.

Use a Pasteur pipette for washing with **ice-cold** methanol.

Although this experiment is short, there are a lot of observations to record. Make sure to watch for colors (of both liquids and solids), especially color changes, cloudiness (or transparency), heat evolution, and amounts of solvent for recrystallization.

We will analyze the product using IR and melting point.

Problems

From the book- chapter 28, questions 1, 2. With question 1, do not just give an equation. You must explain **why** the material dissolves (consider how sulfuric acid will behave as a solvent). Also:

- How is unreacted methyl benzoate removed from the product? Give at least two places in the procedure.
- Write a mechanism for the formation of the nitronium ion from nitric & sulfuric acids.
- Assign the peaks in the ^1H NMR spectrum for methyl 3-nitrobenzoate (p370).