

Lab 06: Nitration of Methyl Benzoate

Reading

- Pavia 3rd edition, et al., Expt. 40, pp. 342–346.

Prelab Assignment:

Your prelab should include: Date, Title, Introduction, Haz-Mat, Apparatus, Chemical Equation, Reactant Table, and Procedures.

Procedural Notes

We will follow the procedure in the lab book. Please note the following:

1. Remember to always stand the conical vials in a small beaker to avoid them falling over and spilling.
2. Measure the methyl benzoate with a syringe and needle. Do not pierce the septum on the bottle.
3. Use the provided glass measuring serological pipettes to measure the sulfuric acid and nitric acid. Please do not mix up the pipettes.
4. The methyl benzoate and the sulfuric acid must be completely mixed (homogeneous) before the $\text{HNO}_3/\text{H}_2\text{SO}_4$ mixture is added. The stir bar might not be sufficient to mixture the reaction. If not, mix by gently shaking the conical vial back and forth, or mix using a glass stir rod.
5. After addition of the $\text{HNO}_3/\text{H}_2\text{SO}_4$ mixture is complete, let the solution warm to room temperature in a water bath for about 15 min. Then, the mixture should be left for an additional 15 min at room temp.
6. The reaction mixture is transferred into ice to dilute the acids and stop (quench) the reaction. Do not weigh the ice; just use about 5 mL volume in a small beaker.
7. The product should precipitate from the ice-water mixture as a white-yellow solid. It may be necessary to vigorously stir the ice-water mixture with a glass stir rod to induce precipitation. Do not vacuum filter the mixture if there is no solid. Instead, ask the instructor for help getting the precipitate to form. Don't forget to weigh the crude product before recrystallizing.
8. Recrystallize the crude product from methanol in a test tube. Use a water bath on the hot plate to heat the test tube.
9. Be careful not to overheat and melt the product. If the product melts, two liquid layers will be seen in the test tube. Continue to add methanol until the bottom layer completely dissolves before cooling and crystallizing.
10. The product should be air-dried before determining the final weight, melting point, and IR spectrum.

Additional Notes

- Sulfuric acid is used twice in the reaction. Make two separate entries in the reaction table for the two separate quantities of sulfuric acid. The larger quantity of sulfuric acid is used as the solvent. Don't calculate its mass or mole quantities for the solvent (the 0.45 mL volume).

Postlab Questions

Answer these questions in your lab notebook after your conclusion is a section titled "Post-lab Questions"

1. Draw the mechanism for the nitration of methyl benzoate.
2. Why is methyl benzoate a meta-director?
3. Why is it important to add the $\text{HNO}_3/\text{H}_2\text{SO}_4$ mixture slowly over a 15 min period?
4. Draw the structures of possible side product(s) that could be formed in this reaction.
5. Write the nitration products for the following reagents:
 - a. Toluene
 - b. 1,3-dimethylbenzene
 - c. Benzaldehyde.
6. Predict the effect on each of the following if the product was not completely dried after the recrystallization:
 - a. percent yield
 - b. melting point
 - c. IR spectrum.