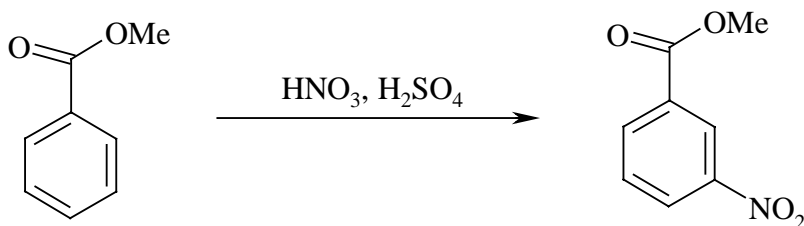


The Nitration of Methyl Benzoate



Procedure:

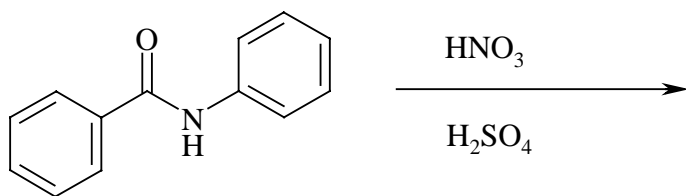
1. Put 0.6 ml of H_2SO_4 in a reaction tube and cool in an ice bath (0°C). Add 0.30 g of methyl benzoate, mix well and cool again to 0°C .
2. In a separate beaker, mix 0.2 mL of concentrated sulfuric acid and 0.2 mL of fresh concentrated nitric acid and cool the mixture to 0°C . Add the mixture slowly to the reaction tube (with the methyl benzoate) keeping the tube cool. Do not let the reaction rise above 15°C .
3. After all the nitric acid has been added, mix well and warm the mixture to room temperature. After 15 min., pour the mixture onto approximately 2.5 g of ice in a small beaker. A solid should form.
4. Isolate the solid by filtering through a Hirsh funnel. Rinse the solid in the Hirsh funnel with water and a tiny amount (0.2-mL portion) of ice-cold 50 % methanol (mix 1:1 methanol-water). If the wash methanol is not ice-cold, product can be lost in this washing step. Weigh the product. Save a small sample for melting-point determination and analysis by thin-layer chromatography.
5. Recrystallize from methanol. You should need very little methanol (~0.3 ml). Allow the product to dry over the week and find a final yield and melting point.

Notebook: Don't forget to prepare your notebook as stated in previous materials and don't forget to write down the actual amounts used.

Report: Do not forget to include 1) % yield 2) Rf's 3) Theoretical yield and calculations based on actual amount of starting material 4) 4 errors and a modification.

Post Lab Questions

1. Please write a mechanism for the formation of NO_2^+ from nitric and sulfuric acid.
2. Please write a mechanism for the reaction of NO_2^+ with methyl benzoate.
3. In the experiment, an excess of nitric acid was used. Given that the nitro group is an electron-withdrawing group, explain why your reaction stopped with only single nitration but didn't give double nitration.
4. If the reaction were heated for some time, di-nitration products would be produced slowly. What would be the product of the di-nitration of methyl benzoate?
5. There is only one product expected from the mono-nitration of the following compound. What is the product? (Consider the nature of substituents on each of the rings.)



6. The following page shows an NMR and IR for your product. Please explain these spectra using the NMR and IR report form (<http://web.fccj.org/~smilczan/ten/NMRguide.doc>).

