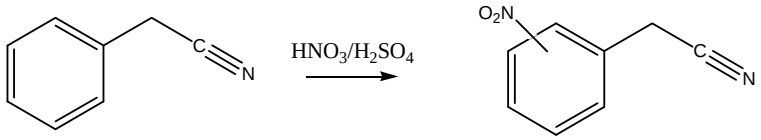
Nitration of Phenylacetonitrile

Question: Is the $-CH_2CN$ group an *o,p* director or a *m*-director? Does the nitration of phenylacetonitrile lead to the *ortho, meta,* or *para* product as the major product?

Reading: Read up on electrophilic aromatic substitution (specifically nitration of benzene) in your organic textbook (Klein, Chapter 19, section 19.4, mechanism 19.5). Also, read up on activating vs. deactivating groups, *o,p* vs. *m*-directors (Klein, Chapter 19, sections 19.7-19.10).

Background: Electrophilic aromatic substitution allows us to add a variety of functional groups directly to a phenyl ring. Such reactions include halogenation, sulfonation, alkylation, and acylation. In this lab, we will perform a nitration, or addition of the $-NO_2$ substituent.



If an aromatic compound has one substituent, nitration could potentially occur at the *ortho*, *meta*, or *para* position (giving a 1,2-, 1,3-, or 1,4-product.) The identity of the major product is controlled by the identity of the original substituent on the substrate; some groups are said to be *o,p*-directors, while others are *m*-directors. In this experiment, you will determine the identity of the major product of the nitration of phenylacetonitrile and use this data to label the $-CH_2CN$ group as either an *o,p*-director or *m*-director.

Procedure: In a small Erlenmeyer flask cooled in an ice bath in the hood, 3 mL of sulfuric acid is added slowly to 3 mL of concentrated nitric acid. Phenylacetonitrile (1.0 mL) is added to the acid dropwise with a Pasteur pipet over five minutes. (Gently and continuously swirl the flask in the icebath during the addition to maintain a low temperature.) After the addition is complete, remove the flask from the icebath and allow to stand at room temperature for one hour. Slowly pour the solution onto crushed ice in a beaker (~40g) and stir with a glass rod until the ice is melted. Filter the solid that forms by suction filtration and wash the product well with cold water. Transfer the crude moist solid to a small beaker and add a mixture of 5 mL ethanol/2 mL distilled water. Heat the covered beaker with stirring until the solid dissolves, then allow the product to recrystallize. Filter the product, pull air through the product until dry, and obtain a melting point and IR. Collect all filtrates as waste in a container in the waste hood.

Results – Calculate your yield and tabulate all of the characterization data that you used to identify your product. Your spectra should be taped into your lab notebook, and your tables should reference the page on which the spectra are taped so that your table data can be easily compared to the raw data in the future. Copies of spectra from databases can also be referenced and included in your notebook for comparison.

Comments:
What is the major product formed in this reaction? What data support this claim? Is your IR spectrum helpful in distinguishing ortho, meta, and para products? Does it give evidence that you made the compound you say you made? Based on your data, is the $-CH_2CN$ group electron donating or electron withdrawing? Is it an *o,p*-director or *m*-director? Draw a mechanism for formation of the major product in this reaction and explain why this mechanism is favored over one of the other possibilities.

<u>Lab 7 assignment:</u> Turn in a hardcopy of your lab notebook (carbon copy or photocopy) attached to a full formal report

- Due at the beginning of lab next week
- 50 pts based on in-lab performance, completion of all sections, correctness of content
- Find ONE paper using SciFinder Scholar. Use it as a reference in your introduction section.
- Submit an electronic copy through Turnitin.com prior to your lab section, or it will be considered LATE