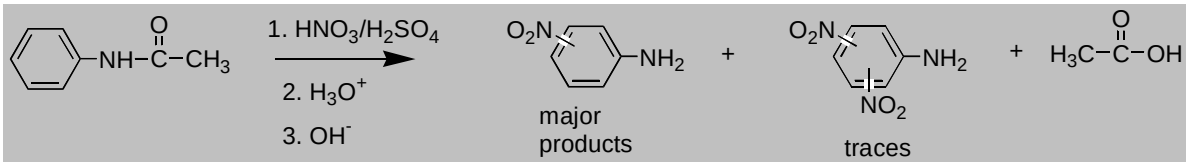
REAC 716: Nitration of Acetanilide and Separation of Nitroanilines



1. Dissolve **2.7g** of acetanilide in **5mL** of H_2SO_4 in 125 mL Erlenmeyer flask. Heat carefully, if needed. Cool flask on an ice bath

2. IN THE HOOD prepare nitrating mixture: measure **1.5mL** of HNO_3 in 10mL graduated cylinder, and then add slowly **4mL** of H_2SO_4. Swirl during the addition. Cool nitration mixture on an ice bath

3. Add nitrating mixture, portion wise, to the solution in 125mL flask. Swirl every time. Flask should not become warm to touch. Let flask stay in ice bath for 15 min

4. Swirling the flask, carefully add to the flask 25mL of cold water from ice bath. A mixture of nitroacetanilides will precipitate.

5. To hydrolyze anilides, cover mouth of flask with aluminum foil and heat the mixture on boiling water bath, until precipitate dissolves. Cool solution in an ice bath.

6. IN THE HOOD, add to the flask 25mL of 25% aq. NH3 solution, in ~5mL portions (exotermic reaction!) Cool the mixture on an ice bath. Vacuum filter crystals

7. Recrystallize the product from minimum amount of 95% ethanol (~10 mL). Collect and dry crystals of nitroaniline. Save mother liquor

8. Take TLC of crystals and mother liquor, using methylene chloride as an eluent.

9. Take IR and mp of crystals. Weight crystals and calculate % yield.

10. Submit IR-spectrum, and TLC-plates. Turn in product in a labeled vial.

11. Submit 5 mg of your nitroaniline product in NMR-tube. Label the tube.

All solutions go to one WATER-BASED waste bottle.