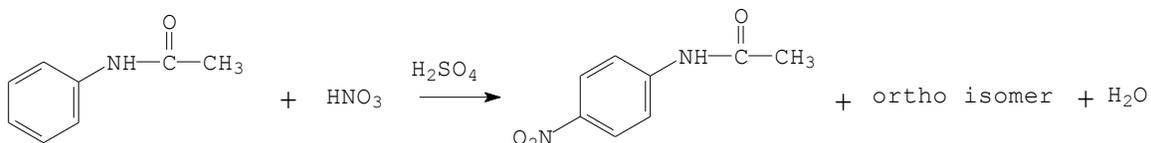


Student Name/ ID \_\_\_\_\_

**Synthesis of *p*-Nitroacetanilide  
(Electrophilic aromatic substitution)**

In this experiment, we convert acetanilide to *p*-nitroacetanilide.



The mechanism for the nitration is that of electrophilic aromatic substitution. The nitronium ion is directed to the positions ortho and para to the acetamido (-NHCOCH<sub>3</sub>) group. This occurs because the resonance electron-releasing effect of that group increases the electron density at those positions, helping to stabilize the intermediates that are formed. Substitution para to the acetamido group is favored over substitution ortho to that group, because the great bulk of the acetamido group shields the ortho positions from approach by reagents. This steric hindrance makes ortho substitution much less likely than para substitution, in which the bulk of the acetamido group has no influence. The ortho substitution product is formed in small quantities in this reaction.

**SPECIAL INSTRUCTIONS:**

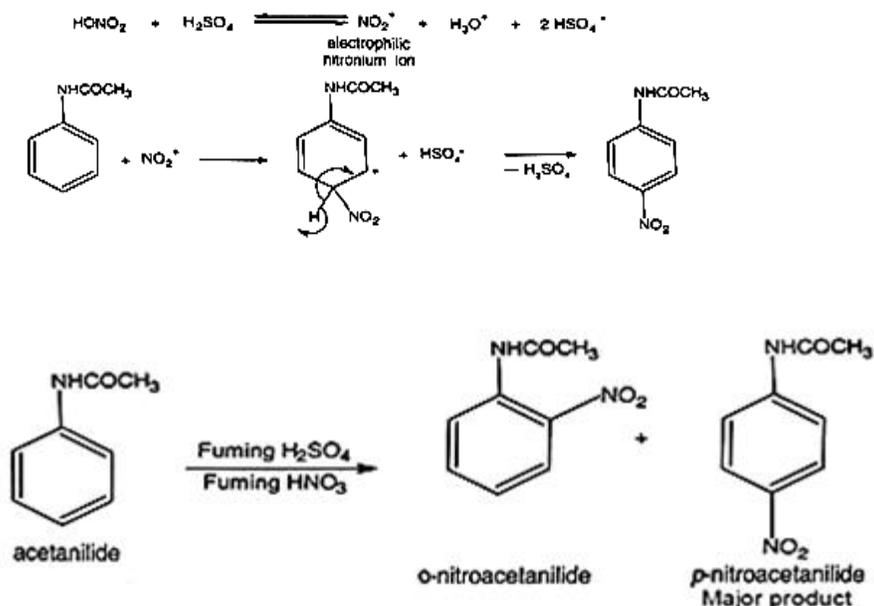
Concentrated sulfuric and nitric acids, in combination, form a very hazardous and corrosive mixture. These acids should be poured together carefully, and this procedure should be carried out in a hood, since noxious vapors are produced. You will begin with the sample of acetanilide you isolated and purified in the first two experiments. If you do not have the required 1.5 grams of acetanilide, you may obtain more acetanilide from the instructor. As always, avoid contact of all chemicals with skin, eyes, and clothing. Wash all contact areas with large quantities of water.

**Chemicals:**

1. Acetanilide
2. Conc. sulphuric acid
3. Nitric acid
4. Distilled water

**Glassware and apparatus:**

Analytical balance, conical flask, glass rod, beaker, measuring cylinder.

THEORYPROCEDURE

- 1- Place 1.5 g of acetanilide in a 125-mL Erlenmeyer flask. Add slowly about 2.5 mL of concentrated sulfuric acid to the acetanilide.
- 2- Dissolve most of the solid by swirling and stirring the mixture. Do not be concerned if a small amount of undissolved solid remains. It will dissolve in later stages of this procedure. Place the flask in an ice bath.
- 3- Place 0.9 ml of concentrated nitric acid in another small flask and add about 2.5 ml of concentrated sulfuric acid to it.

**CAUTION: This is a hazardous mixture. This mixing should be done carefully in a hood. Mix the acids thoroughly.**

- 4- Using a disposable capillary pipet, add the mixed acids **dropwise** to the cooled sulfuric acid solution of acetanilide.
- 5- After each addition of acids, swirl the mixture thoroughly in the ice bath. Do not allow the flask to become warm to the touch. After 20 minutes, including the time required for adding the nitric acid-sulfuric acid mixture, add 12.5 mL of an ice-water mixture to the reaction mixture.
- 6- A suspension of nitroacetanilide isomers will result. Allow this mixture to stand for 5 minutes, with occasional stirring
- 7- Isolate the crude solid by vacuum filtration and recrystallize this solid from water. Allow the crystals to air dry. When the crystals are dry, weigh them and obtain a melting point.

Calculations and results:

Here limiting reagent is acetanilide; hence yield should be calculated from its amount.

Molecular formula of acetanilide =  $C_8H_9NO$

Molecular formula of p.nitroacetanilide =  $C_8H_8N_2O_3$

Molecular weight of acetanilide = 135 g/mole

Molecular weight of p.nitroacetanilide = 181g/mole

*Theoretical yield:*

135 g acetanilide forms 181 g

Therefore, 1.5 g acetanilide will form .....? (X) g p.nitroacetanilide

$$X = (181 \times 1.5) / 135 = 2.01 \text{ g}$$

Theoretical yield = 2.01 g

Practical yield = \_\_\_\_\_ g

$$\% \text{ Yield} = (\text{Practical Yield}) / (\text{Theoretical Yield}) \times 100$$

$$\text{Practical Yield} = \text{_____} \times 100$$

**2.01**