Multistep Azo Violet Procedure

Day 1:

1. Add 3.6 mL of aniline to a 250 mL Erlenmeyer containing 100 mL of 0.4 M HCl and a stir bar.
2. Warm the solution to about 50 °C.
3. Prepare a solution of 6 g sodium acetate trihydrate in 20 mL of distilled water.
4. Add 4.4 mL of acetic anhydride to the warm solution of aniline, then immediately and the sodium acetate solution.
5. Cool the reaction in an ice-water bath for about 10 minutes.
6. Filter the solid product using a Buchner funnel and let the product air dry until the next lab period.

Day 2:

1. (Remember to weigh the dried product from Day 1, get the melting point, and obtain an IR spectrum.)
2. Put 1 g of the acetanilide prepared during Day 1 in a 25 mL Erlenmeyer flask and add 5 mL of glacial acetic acid and your one inch stir bar.
3. While stirring, carefully add 5 mL of concentrated sulfuric acid.
4. Once the solid has dissolved, cool the solution in an ice-water bath.
5. Prepare a solution of 2 mL concentrated nitric acid in 1.3 mL of concentrated sulfuric acid.
6. Very slowly (i.e., dropwise) add the acid mixture to the reaction flask. Once the addition is complete, let the reaction stir at room temperature for 20 minutes.
7. Pour the reaction mixture into a beaker containing 25 mL of cold water and 15 g of ice (ie, a 40 mL ice water solution).
8. Filter the solid product using a Buchner funnel and wash the product 3x with water.
9. Recrystallize the product with ethanol.
10. Filter the solid product using a Buchner funnel and let the product air dry until the next lab period.

Day 3:

1. (Remember to weigh the dried product from Day 2, get the melting point, and obtain an IR spectrum.)
2. Add 0.74 g of the *p*-nitroacetanilide to 5 mL of water in a 25 mL round-bottom flask along with your one inch stir bar.
3. While stirring, slowly add 5 mL of concentrated sulfuric acid, attach a condenser and gently boil the solution for 30 minutes.
4. Let the solution cool and then pour the reaction mixture into a beaker containing 20 mL of cold water and 15 g of ice (i.e., a 35 mL ice-water solution).
5. While stirring, add 9 M sodium hydroxide until the pH of the solution reaches 4-5.
6. Cool the mixture, then filter the solid product using a Buchner funnel and let the product air dry until the next lab period.

Day 4:

1. (Remember to weigh the dried product from Day 3, get the melting point, and obtain an IR spectrum.)
2. In a 25 mL Erlenmeyer flask, prepare a solution of 0.072 g of *p*-nitroaniline made during Day 3 in 2.7 mL of concentrated HCl and 10 mL of water. Place the solution in an ice-water bath.

3. Once cooled, add a solution of 0.035 g sodium nitrite in 1.5 mL of water dropwise while stirring. Continue stirring in the ice bath for 10 minutes.
4. Prepare a solution of 0.055 g resorcinol in 9 mL methanol in a 125 mL Erlenmeyer flask and place the solution in an ice-water bath.
5. While stirring, add the sodium nitrite solution slowly. Continue to stir in an ice-bath for 30 minutes.
6. Neutralize the solution with sodium acetate until the pH of the solution is 5-6. Stir for 30 minutes at room temperature.
7. Filter the solid product using a Buchner funnel and wash with water.
8. Perform the dyeing test and indicator tests.

Dyeing test:

1. Add a scoop of the product to a 50 mL beaker, 0.5 mL of 1 M sodium sulfate, 15 mL water and 5 drops of 1 M sulfuric acid.
2. Heat the solution to boiling and place a piece of cotton in the beaker for 2 minutes.
3. Remove the cotton and record the color.

Indicator test:

1. Add a small spatula tip full of the azo violet product to 3 test tubes.
2. Add a few drops of 0.5 M HCl to the first tube and a few drops of 0.5 M NaOH to the second tube.
3. Record the color of each solution.