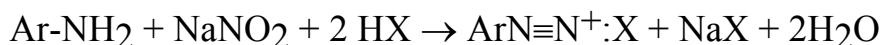


Diazotization: Synthesizing A Dye

Introduction

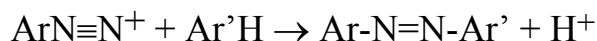
Dyes have been used for centuries to color clothing and skin. Dyes are also used to determine the pH of different solutions, e.g., phenolphthalein (a pH indicator). In addition, dyes are used to stain bacteria and other micro-organisms to identify them.

There are a number of types of dyes. This experiment focuses on the synthesis of a diazo dye, Orange II. The first step in the preparation of Orange II is the preparation of a diazonium salt. In general (and "Ar" = an aromatic group), the preparation is as follows:



Diazonium salts ($\text{ArN}\equiv\text{N}^+:\text{X}$) are very unstable – even on ice – and must, therefore, be used as soon as they are generated.

The second step, then, in the production of a diazo dye is coupling the diazonium salt with a phenol:



The phenol which is attached by the diazonium salt MUST contain strong ortho-para director (what are three examples of these? Meta directors?). Electrophilic aromatic substitution generally occurs para to a directing group – there are exceptions, Orange II being one.

Materials and Methods

Materials

In the table, below are the necessary chemicals and supplies for this experiment:

Sulfanilic acid	β -naphthol	Buchner funnel and filter paper	Spatula
Anhydrous sodium carbonate	Glass stirring rod	Warm water bath	Heat lamp
Sodium nitrite	NaCl-- solid and cold sat'd solution	250 mL beaker	400 mL beaker
Concentrated HCl	250 mL Erlenmeyer flask	Ice bath	10% NaOH (place on ice before experiment)

Method

Reaction 1: Place 4.8 grams of sulfanilic acid monohydrate, 1.33 grams of anhydrous sodium carbonate and 50 mL water in your 250 mL Erlenmeyer flask. Warm it gently in the water bath with swirling until there is a clear solution. Then cool the solution on the ice bath for 15 minutes. To this cooled solution, add 5 mL of a solution of sodium nitrite in water (1.9 grams NaNO_2 in 5 mL water). Mix, then pour the whole solution, with stirring, into a 250 mL beaker containing around 30 grams of crushed ice and 5 mL concentrated HCl. You will see a fine white precipitate forming very quickly. This is your diazonium salt. The whole mixture will be used in the next portion of the reaction.

NOTE: The sodium carbonate makes the sulfonic acid water-soluble.

Reaction 2: Mix 3.6 grams of β -naphthol with 20 mL of cold 10% NaOH (if necessary, you can use 10% KOH) in a 400 mL beaker. To this solution, add the well-mixed and cooled suspension from Reaction 1. Stir well. The dye formation occurs rapidly and will separate from the rest of the mixture. Continue stirring for 10-15 minutes to complete the reaction, then heat the mixture until as much of the solid has dissolved as possible. To the warm solution, add 10 grams solid NaCl and reheat until it is dissolved. After the NaCl is dissolved, place the beaker of your mixture in the ice bath and allow it to precipitate UNDISTURBED until it is cold. Once the mixture is cold (about 10-15 minutes), stir well and filter through the Buchner funnel. Rinse your dye with a little cold saturated saline, let the moisture pull through, then place the resulting dye with its filter paper on a paper towel and place in the incubator to dry.

NOTE: The NaCl “salts out” the dye, making it less water-soluble so it will crystallize as it cools.

Results

Describe in the space below your observations as you performed this experiment:

Questions

- 1) Write out all of the reactions which occurred as you prepared the dye.

2) Find another Diazo dye and diagram its synthesis: