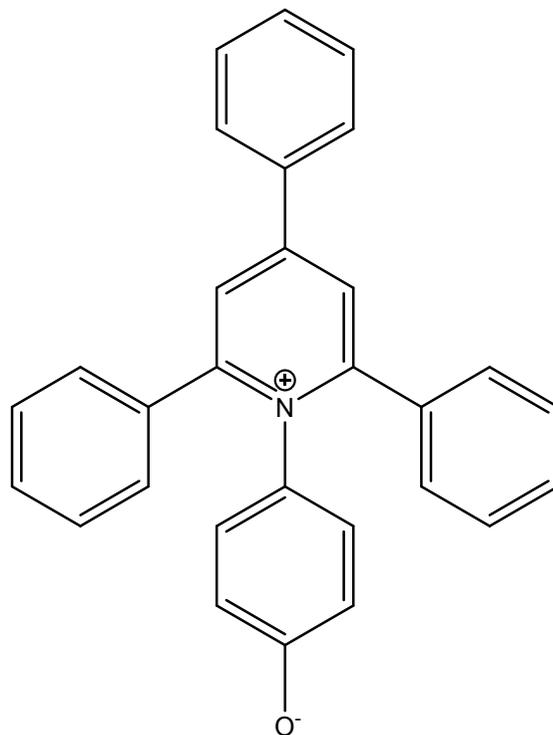


Multi-Step Synthesis of Betaine-30



Name: Betaine-30
Molar Mass: 551.675 g
Melting Point: 200-275 °C

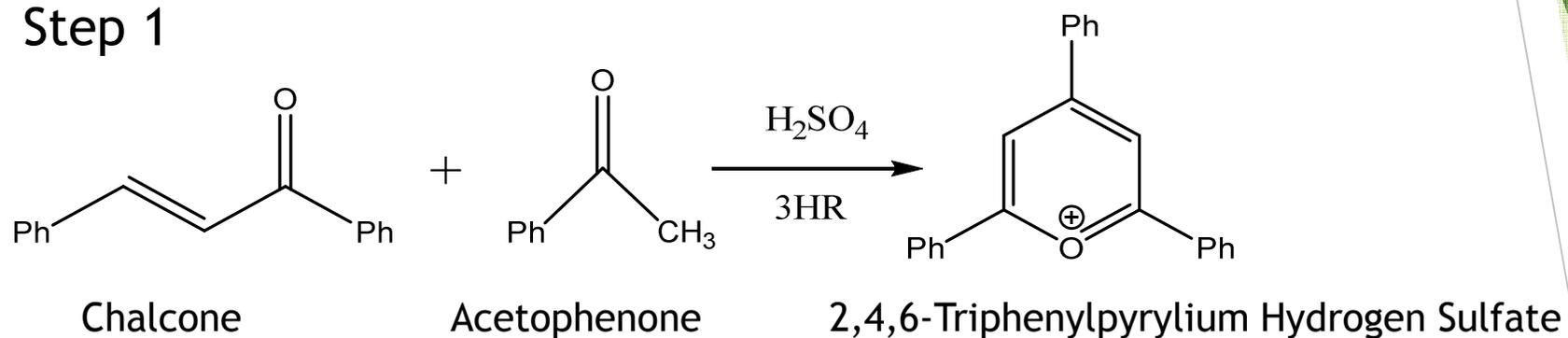
What is Betaine-30?

- ❖ 2,6-diphenyl-4-(2,4,6-triphenylpyridinio)-phenolate (Reichardt's dye)
- ❖ Solvent Polarity Indicators
- ❖ A Solvatochromic Dye
(Ex: Red in methanol, Violet in ethanol, Blue in isoamyl alcohol, green in acetone, and yellow in anisole)

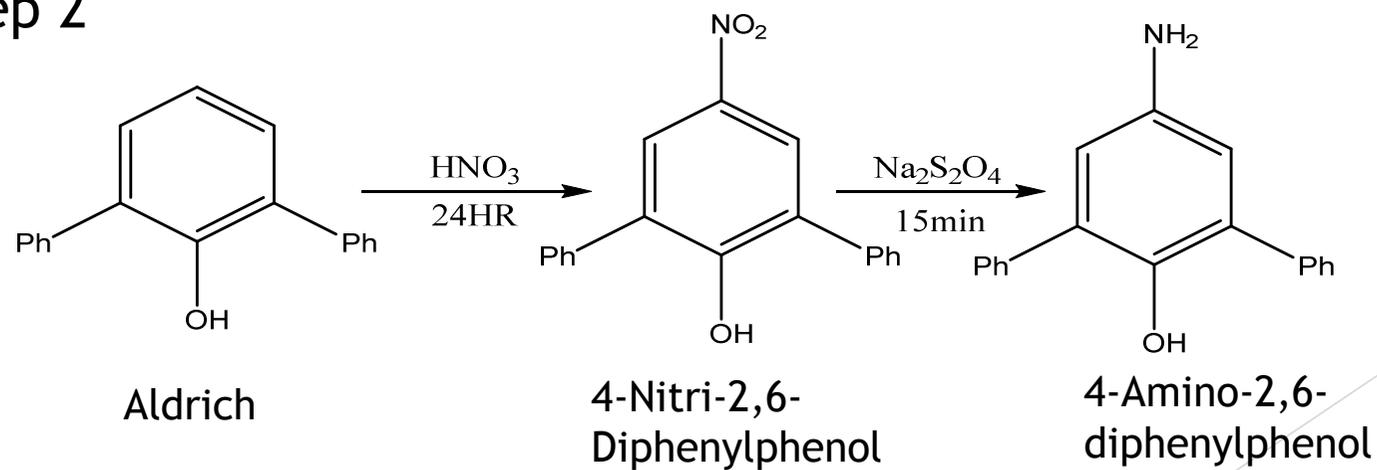


Convergent Synthesis of Betaine-30

Step 1

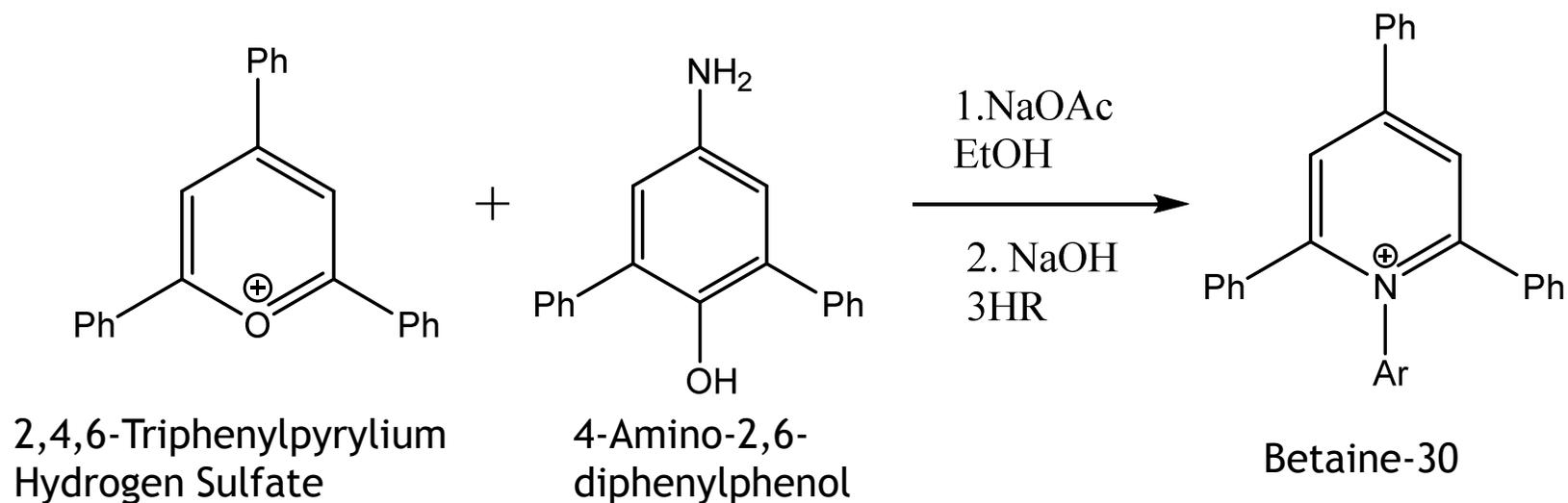


Step 2



Convergent Synthesis of Betaine-30

Step 3



Step 1

Data & Observations

- ▶ 4.28 g (0.0206 mol) of Chalcone, 1.24 g (0.0103 mol) of Acetophenone and 3.2 g of H₂SO₄ was mixed together and heated on the water bath for 3 hours
- ▶ After 3 hours of heating, 20 ml of water was added. A precipitate formed and dissolved on further heating
- ▶ In the process of heating, a black oil separated, and was removed by vacuum filtration
- ▶ A yellow crystal was formed
- ▶ Theoretical Yield: 4.1896g Actual Yield: 1.635 g Percent Yield: 39 %
- ▶ MP: 230 °C (lit.271-273.5)

Step 2a. 4-Nitro-2, 6-Diphenylphenol intermediate

Data & Observations

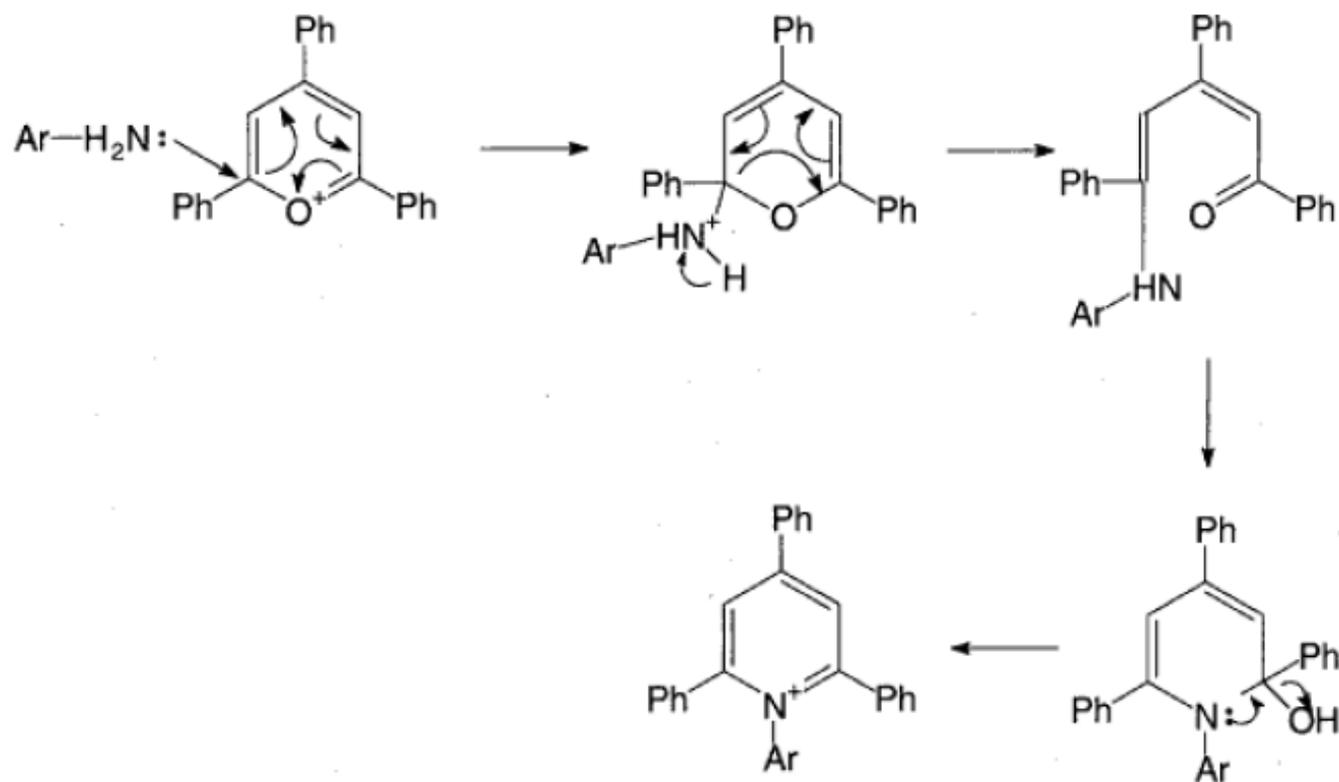
- ▶ 65% HNO₃ and 2.00 g (8.12 mol) of 2, 6-diphenylphenol was mixed together and stir over night
- ▶ Light pink->light yellow->light orange->orange
- ▶ Crude product: 2.717g (orange powder)
- ▶ Mp: 117~120 °C
- ▶ The crude produce was dissolved in hot ethanol and treated with activated Carbon. After vacuum filtration the solution was concentrated by evaporation
- ▶ Purified product: 1.427 g (orange powder)
- ▶ Theoretical Yield: 2.369g Actual Yield: 1.427 g Percent Yield: 60 %
- ▶ MP: 127 ~128 °C (lit: 135 °C)

Step 2b. 4-Amino-2,6-Diphenylphenol

Data & Observations

- ▶ 1.427 g of 4-Nitro-2, 6-Diphenylphenol was added to 50 ml of hot NaOH
- ▶ The solution was expected to turned into deep-red solution while stirring and add small amount of solid sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) until the solution was turned Yellow, and heated for 15 min
- ▶ Add glacial acetic acid to adjusted the solution to pH5, the product precipitated
- ▶ Theoretical Yield: 1.048g Actual Yield: 0.777 g PercentYield: 74 %
- ▶ MP: 133~134 °C (lit: 147-148 °C)

Mechanism for the convergent step in the synthesis of Betaine-30



Step 3 Betaine-30 (Dye ET-30)

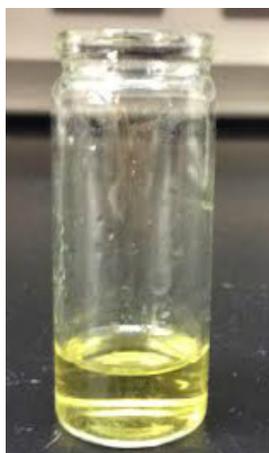
Data & Observations

- ▶ To a small reflux set-up, 2, 4, 6-Triphenylpyrylium Hydrogen Sulfate, 4-Amino-2,6-Diphenylphenol, 0.245 g of anhydrous sodium acetate, and 3.1 ml of ethanol was mix together and reflux for 3 hour
- ▶ After reflux, 1.5 ml of NaOH was added
- ▶ Expecting: Dark blue crystal, and turned green after dry
- ▶ Only get a very little amount of product to form
- ▶ Theoretical Yield: 0.363g Actual Yield: 0.012 g PercentYield: 3 %
- ▶ Wouldn't able to take the MP (lit: 200-275 °C)

Demonstration & Conclusion



Acetone



In other solutions

Although it successfully turned green in Acetone, it was either green yellow or green blue in other solutions (which it supposed be red in methanol, purple in ethanol, blue in isoamyl alcohol, green in acetone and yellow in anisole). Thus, the experiment was a failed

Possible Improvements for Next Time

- Time management
- Clean the Glass wear more carefully (especially stir-bar) !
- NMR and IR

Reference

- **Convergent Synthesis of Betaine-30, a Solvatochromic Dye: An Advanced Undergraduate Project and Demonstration**
Bruce R. Osterby and Ronald D. McKelvey
Journal of Chemical Education **1996** 73 (3), 260