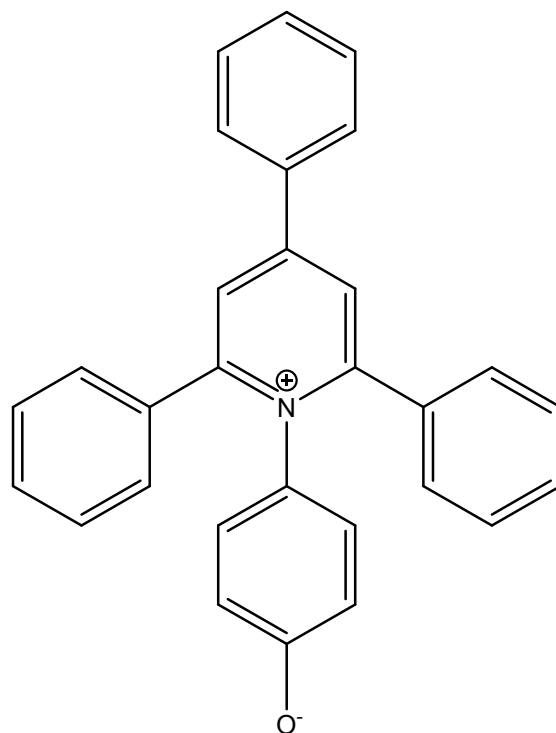


# Multi-Step Synthesis of Betaine-30



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

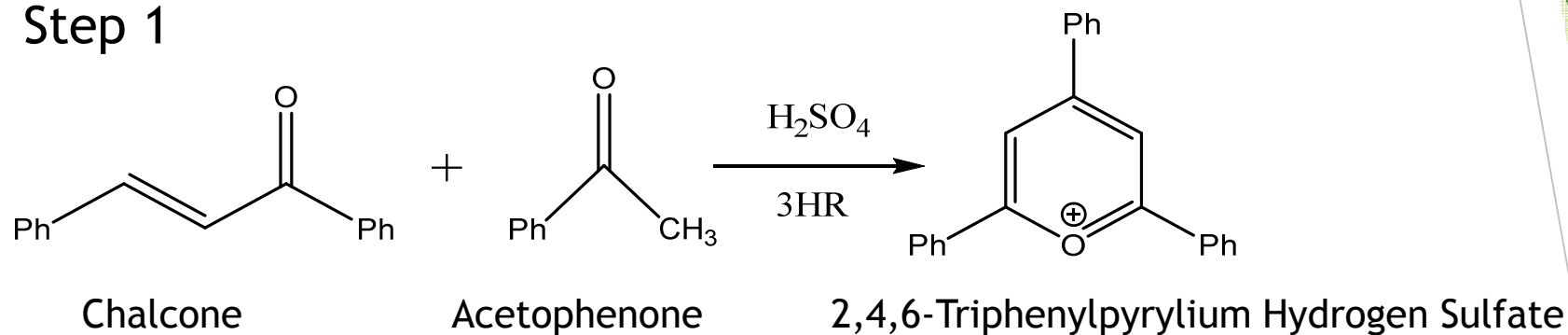
# What is Betaine-30?

- ❖ 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)

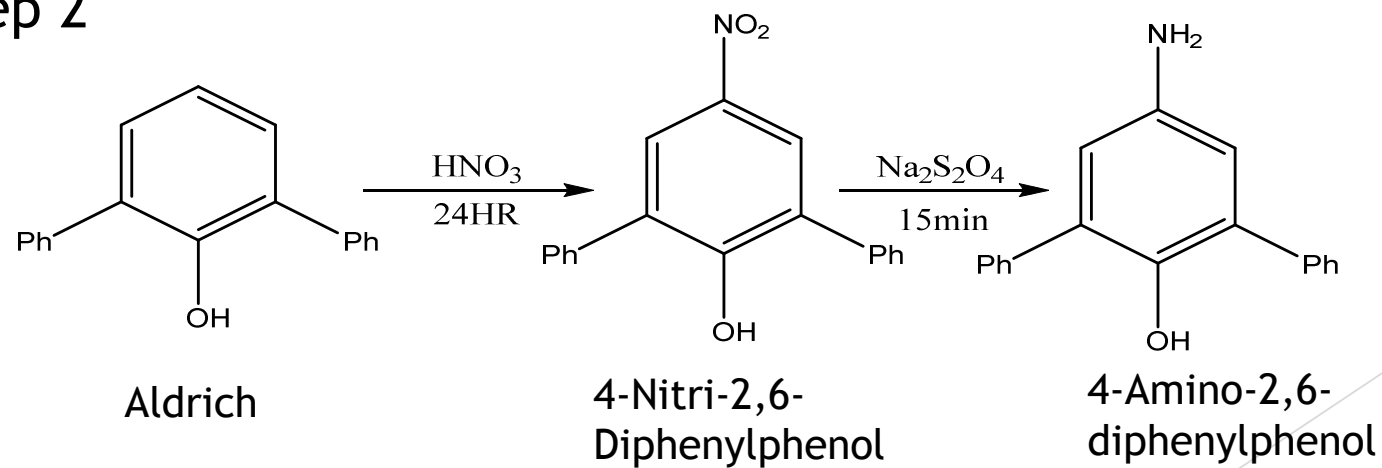


# Convergent Synthesis of Betaine-30

## Step 1

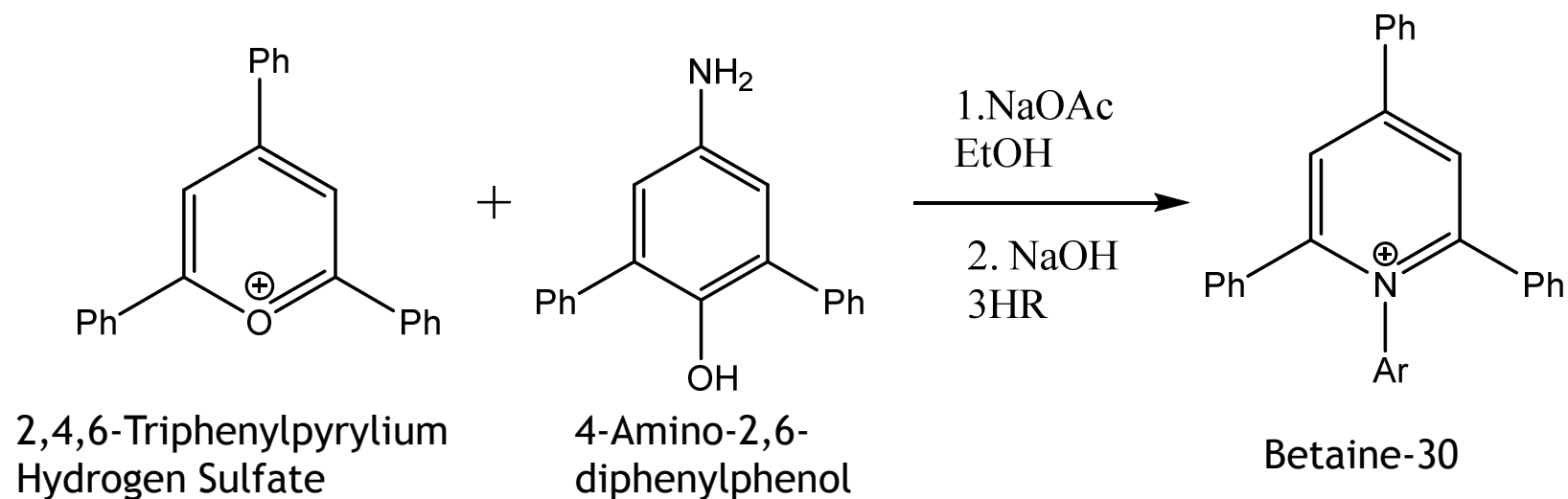


## Step 2



# Convergent Synthesis of Betaine-30

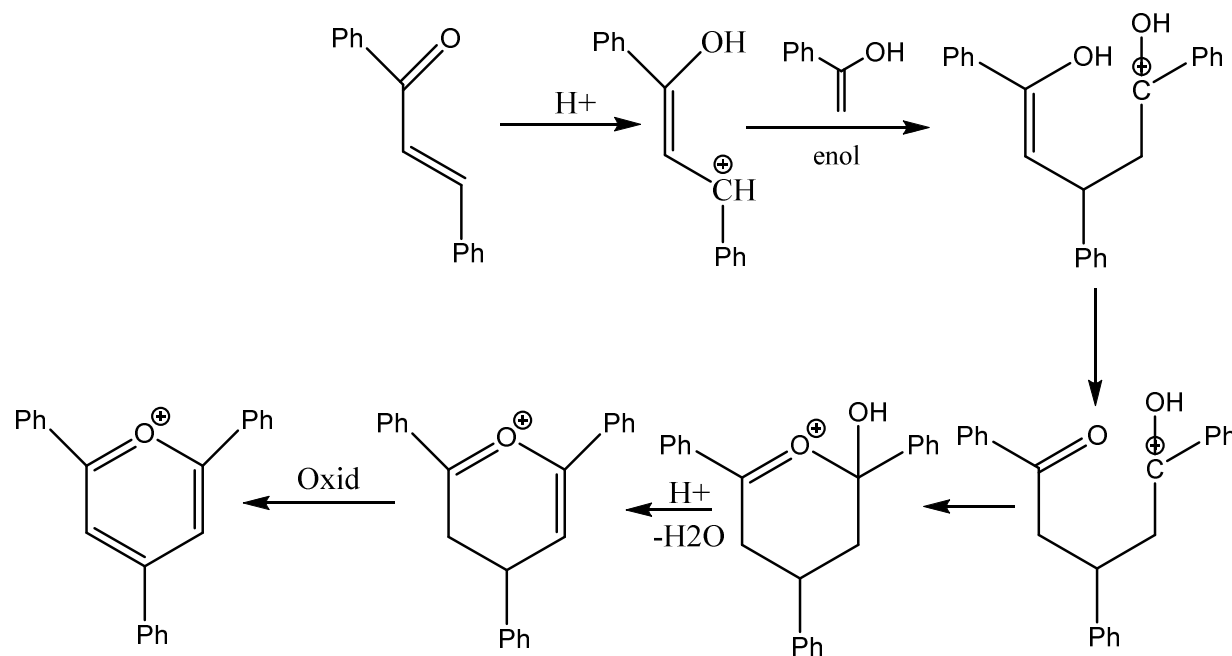
## Step 3





# Step 1

## ( 2, 4, 6-Triphenylpyrylium Hydrogen Sulfate )



Mechanism for the formation of 2, 4, 6-Triphenylpyrylium Hydrogen Sulfate

# 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  $\text{H}_2\text{SO}_4$  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% HNO<sub>3</sub> 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)

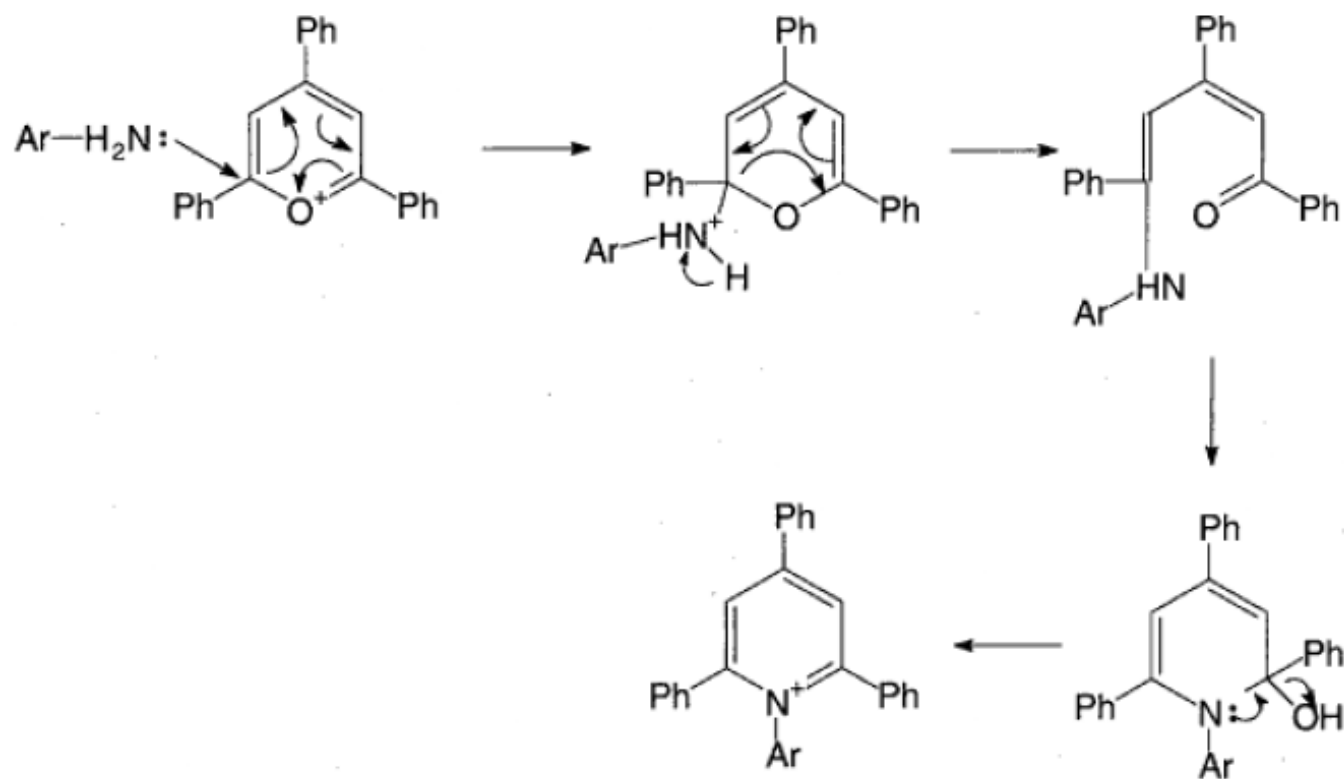
## Step 2b. 4-Amino-2,6-Diphenylphenol

### 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)

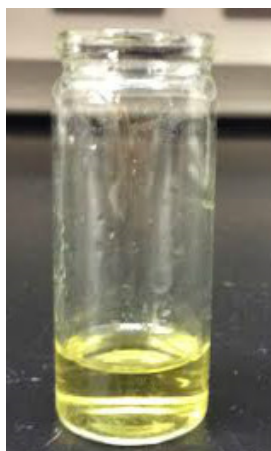
### Data & Observations

- ▶ 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