



# Synthesis of Acid-Base Indicators

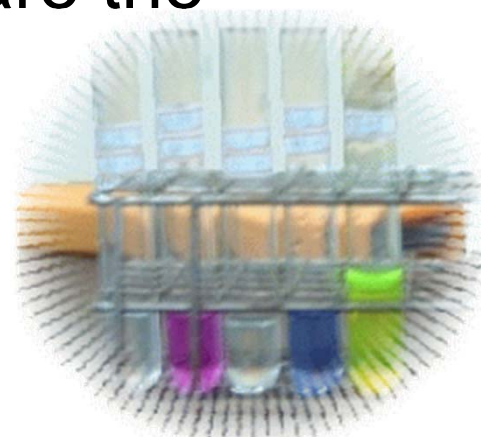
(2016/03/02 revised)

- Clean 6 test tubes to TA and dry in oven
- Collect test tube holder, latex gloves, and dropper
- Concentrated sulfuric acid, phenol and guaiacol are in the fume hood
- Shared instruments: UV light
- Alcohol lamp, wind shield: in fume hood
- **Reactants are corrosive; avoid contacting with skin and eyes**
- **Latex gloves and goggles must be worn at all times**
- **Use test tube holders to hold and carry test tubes**
- **Wash hands after experiment**
- Solid waste must be placed in capped recycling bottles separately to prevent evaporating and inhalation of the vapor <sup>1</sup>



# Objective

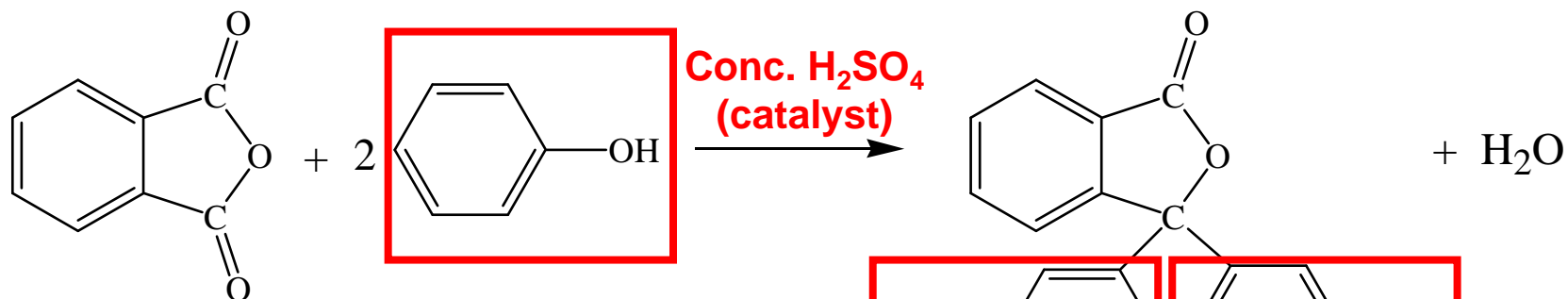
- Learn the chemical structure of the acid-base indicator **phenolphthalein**, its preparation, and the chemistry of its color change
- Change the **substituent** in the structure of phenolphthalein to change its color
- Use the similar procedures to prepare the **fluorescent yellow** and highlighters





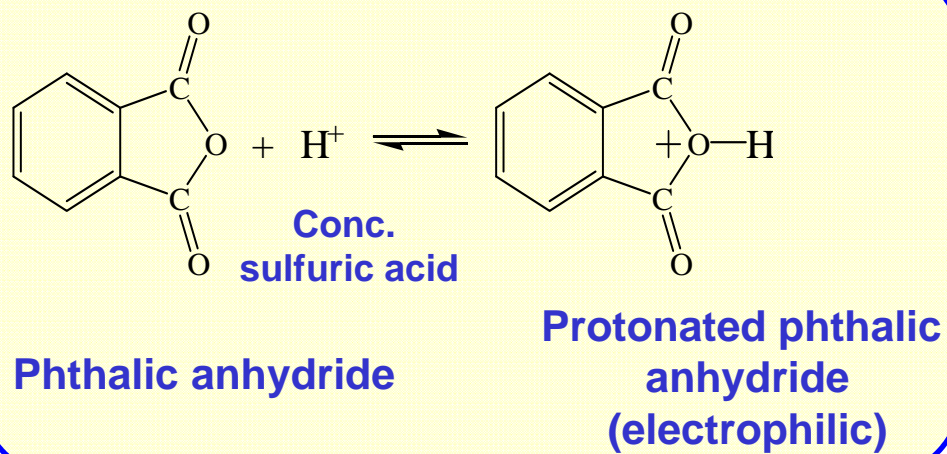
# Preparation of Phenolphthalein

I.



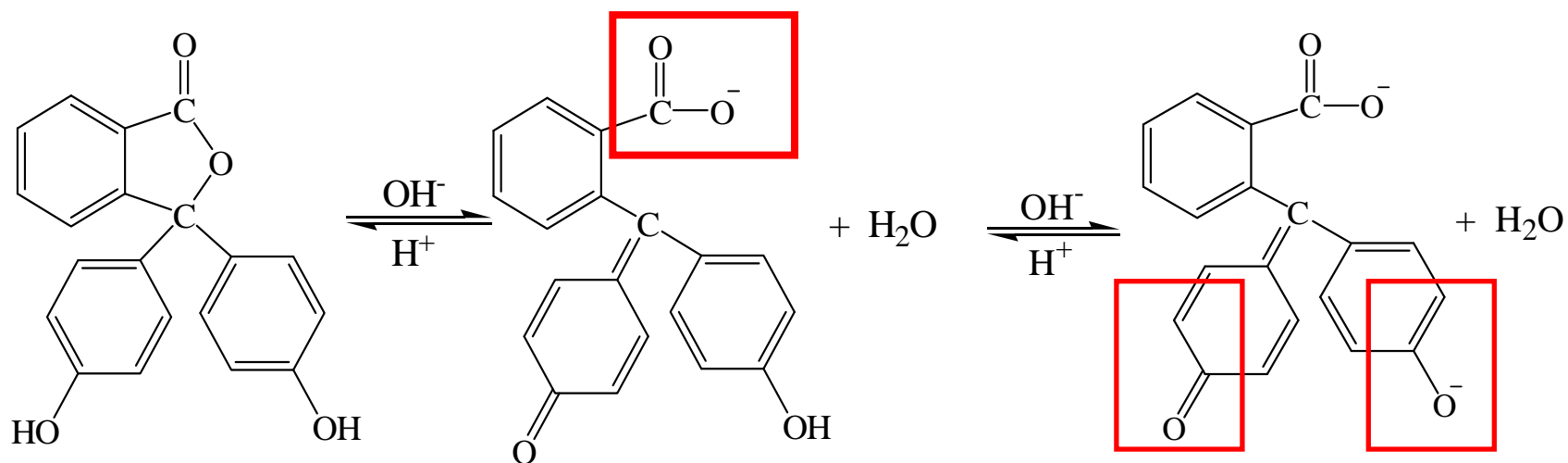
Phthalic anhydride      Phenol

Phenolphthalein





# Color of Phenolphthalein Indicator



**Phenolphthalein in  
acidic soln  
(colorless)**

**Phenolphthalein in  
neutral soln  
(light yellow)**

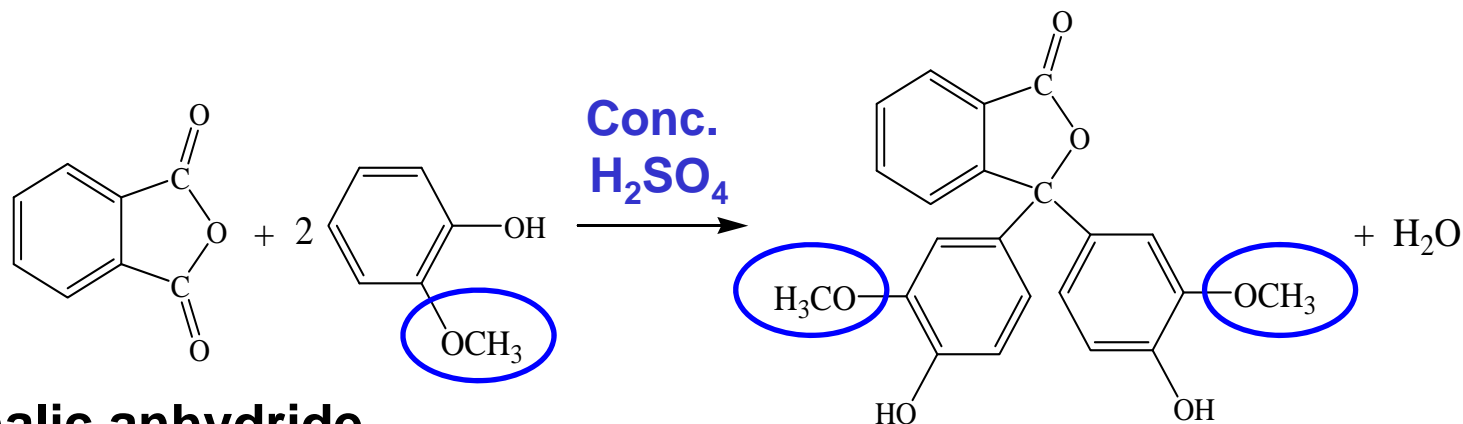
**Phenolphthalein in  
alkaline soln  
(magenta)**





# Effect of Substituent on Color

II.

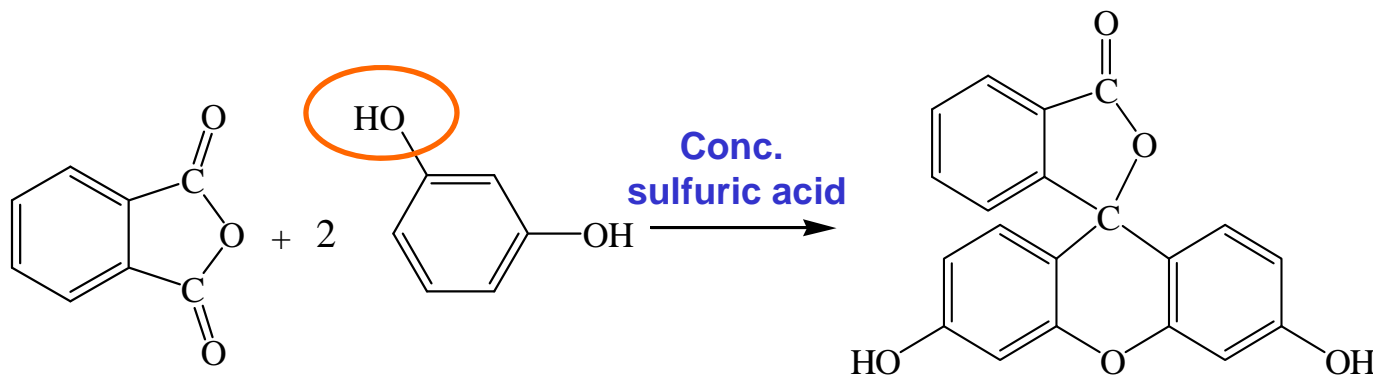


**Phthalic anhydride**

**Guaiacol**

**Diguaiacol phthalein**

III.



**Phthalic anhydride**

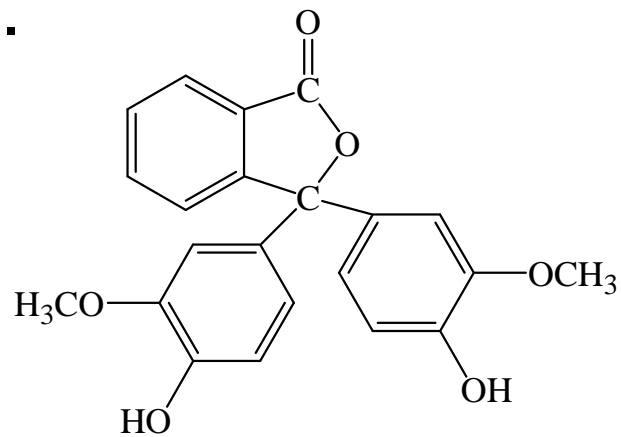
**Resorcinol**

**Fluorescent  
Yellow**

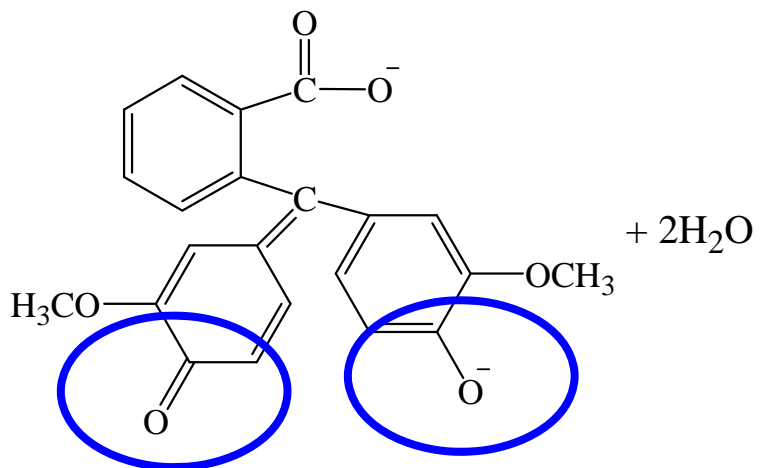
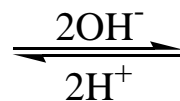


# Color of Diguaiacol Phthalein

II.



Diguaiacol phthalein in acidic soln (colorless)



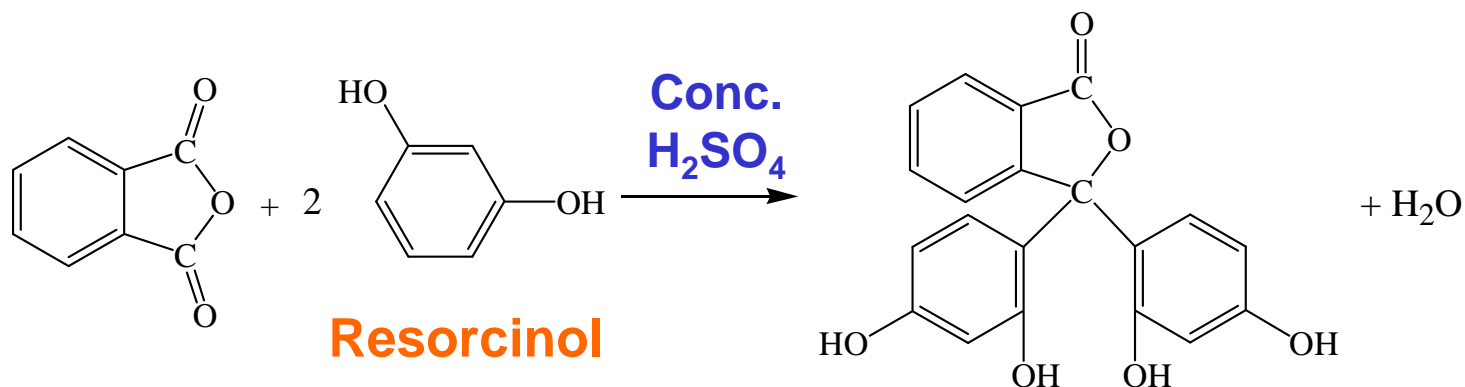
Diguaiacol phthalein in alkaline soln (blue)



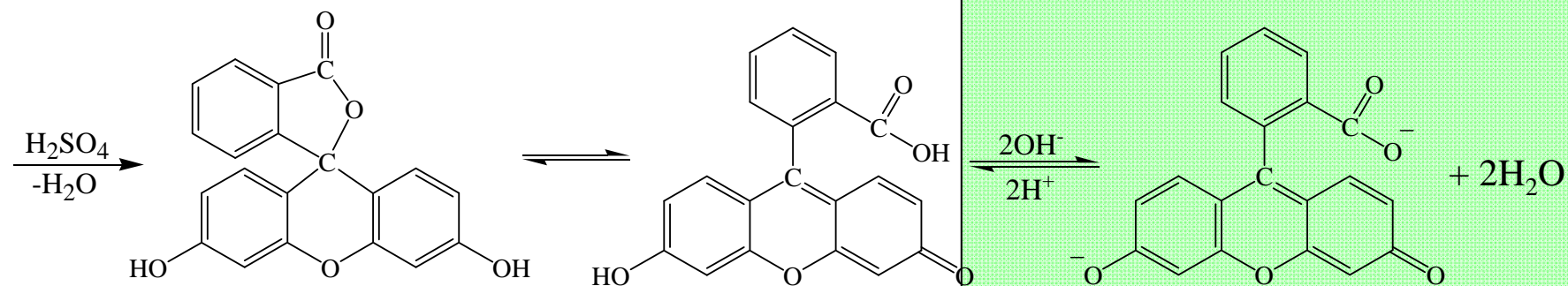


# Synthesis of Fluorescent Yellow

III.



**Phthalic anhydride**



**Fluorescent Yellow**

**Tautomer**

**Green Fluorescing Substance in alkaline soln**

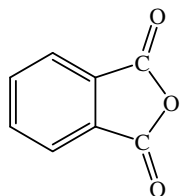


# Flow Chart I

**Operate in hood**

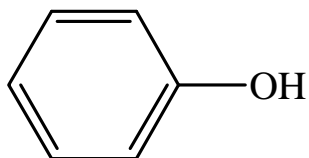
**React**

**Product appearance**

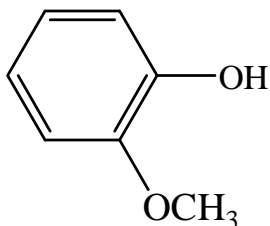


**1/2 spoon  
Phthalic  
anhydride  
+  
2 d Conc.  
H<sub>2</sub>SO<sub>4</sub>**

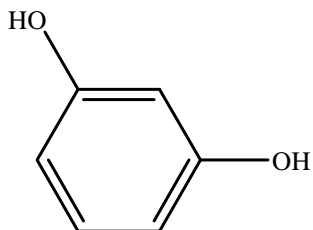
**2 d phenol**



**2 d guaiacol**



**1/2 spoon resorcinol**



**Stir and mix**



**Heat on flame**



**React**



**Color change**



**Solid  
disappear**



**Orange**



**Purple**

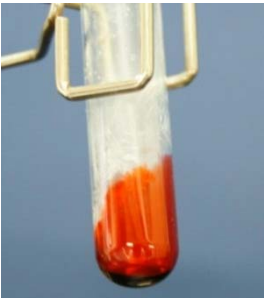
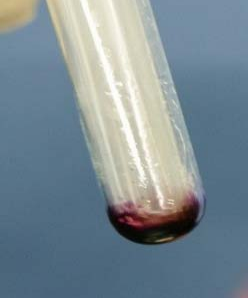



**Dark brown**





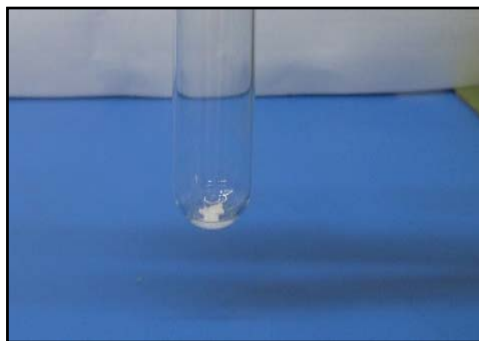
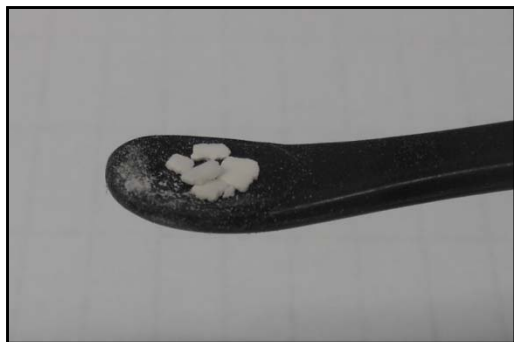
# Flow Chart II

Product from	Add H <sub>2</sub> O in hood	Acid-base test	
 <b>Phenol</b>	Add 3 mL H <sub>2</sub> O ⇓ Stir and mix ⇓	Obtain some solid product ⇓ Dissolve in alcohol	Add NaOH(aq) ⇓
 <b>Guaiacol</b>	Product precipitate out ⇓		Add HCl(aq) ⇓
 <b>Resorcinol</b>	Decant supernatant and get solid product		Observe color change



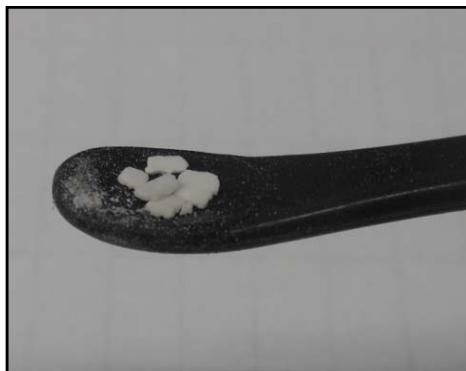
# Preparation

1. One 250 mL beaker
2. One dry test tube with  $\frac{1}{2}$  spoon phthalic anhydride
3. A test tube with 3 mL  $\text{H}_2\text{O}$
4. Glass rod
5. Test tube holder





# Procedure I. Synthesis of Phenolphthalein



**Obtain dry test tube**

↓  
Add 1/2 spoon of phthalic anhydride

↓  
2 d phenol and 2 d conc. sulfuric acid

↓  
**Mix thoroughly with glass rod**

- Phenol and sulfuric acid are corrosive.
- Wear gloves



Heat on flame for 5 s.

↓  
**Move out of flame**

↓  
Stir and heat repeatedly

↓  
Stop heating after all solids have dissolved

↓  
Record color change



Add 3 mL distilled water

↓  
Stir to mix and product precipitate out

↓  
**Decant the supernatant**

↓  
Transfer a portion of solid to test tube



Dissolve with 95% ethanol

↓  
Add drops of 1 M NaOH to observe the color change

↓  
Add drops of 1 M HCl

↓  
Record color change



# Procedure II. Synthesis of Diguaiacol Phthalein



Obtain dry test tube



Add 1/2 spoon of phthalic anhydride



**Add 2-3 d guaiacol** and  
2 d conc. sulfuric acid



Heat to synthesize



Separate products by  
adding water

## Caution:

- Mix the reactants thoroughly
- Heat on flame for 5 s
- Leave out of flame and mix again
- Repeat the operation till color change and solid reactant disappear
- Reaction is faster than phenol and avoid overheating
- Product is sticky with dark blue to purple color



# Procedure III. Synthesis of Fluorescent Yellow



Obtain dry test tube

↓  
Add 1/2 spoon of phthalic anhydride

↓  
**Add 1/2 spoon of resorcinol**

2 d conc. sulfuric acid

↓  
Heat to synthesize

↓  
Separate products by adding water



Obtain a portion of product

↓  
Dissolve with 95% ethanol (soln. A)



Obtain 2~3 d of soln. A into another test tube

↓  
**Dilute with 10% ethanol until light yellow**

## Caution:

**The fluorescent yellow decomposes at 315° C. Test tube should be moved in-and-out of flame to avoid over heating**



# Fluorescence Observation



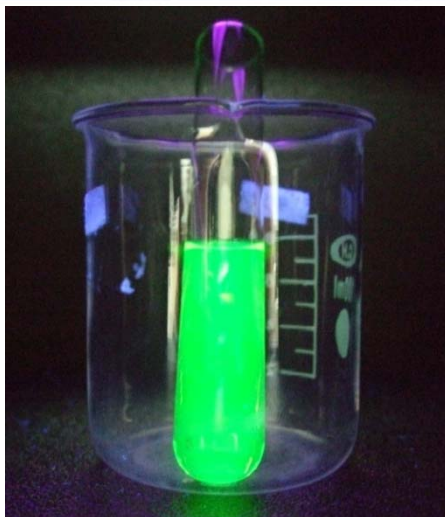
**Add drops of 1 M NaOH**



Use black paper as background



Observe fluorescence under UV lamp (with long / short wavelength)



Fluorescence under UV light



Take portions of soln. A



Add polyvinyl alcohol glue



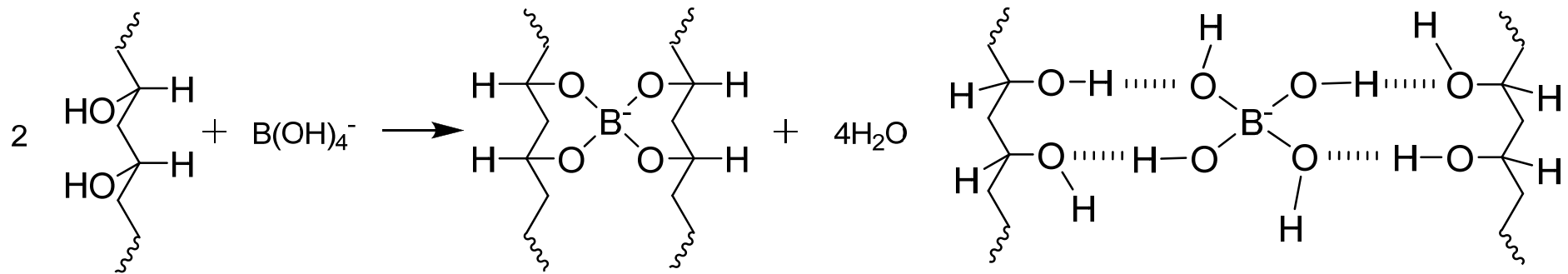
Stir with glass rod to write on paper

**Caution:** Avoid exposing your eyes and skin to UV light



# Fluorescent Plastic

- **The chain of polyvinyl alcohol (PVA) can be cross-linked by borate to form an elastomer**



**Cross-linking with  
covalent bonding**

**Cross-linking with  
hydrogen-bonding**



# Procedure IV. Fluorescent Plastic



- Add 3 d fluorescent yellow (soln. A) in 100 mL beaker
- Add 1 d of 1 M NaOH

- Add 4 mL PVA glue
- Mix with glass rod
- Add 1 mL of sodium borate



- Mix thoroughly with glass rod
- Observe the change in **color** and **viscosity**





# Notice

- **Phenol, conc. sulfuric acid...etc. are corrosive; gloves and goggles should be worn at all times**
- Do not take more than **2 d** of conc. sulfuric acid.
- Limit the use of chemicals: the amount of chemicals used in this experiment does not need to be precise.
- Take small amounts of chemicals into test tube and give out excess chemicals to others
- Mix reactants thoroughly before heating
- Hold test tube with test tube holder, move test tube in and out of flame continuously to avoid overheating
- **Avoid exposing eyes and skin to UV light**
- After experiment, clean test tubes with 10% alcohol, pour the first rinsed waste liquid into recycling bin then clean with water