# Synthesis of Acid-Base Indicators



Last revised: 2025/02/09

# Preparation

## Collect the following items

- One test tube tongs
- Two drop pipets
- NBR gloves
- Shared items:
  - Conc. sulfuric acid, phenol and guaiacol (in fume hood)
  - Hot plate, sand bath, digital thermometer (in fume hood)
  - UV light

## From your personal equipment

- Five test tubes (clean and oven dry)
- Test tube rack, test tubes, glass rod
- 10 mL graduated cylinder
- 100 and 250 mL beakers

# Objective and Lab Techniques

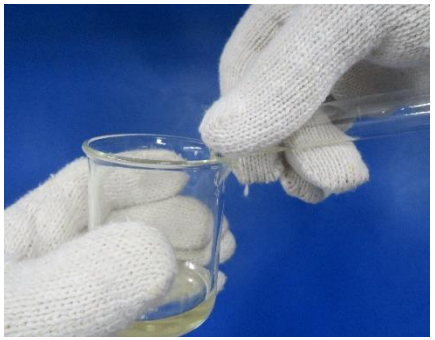
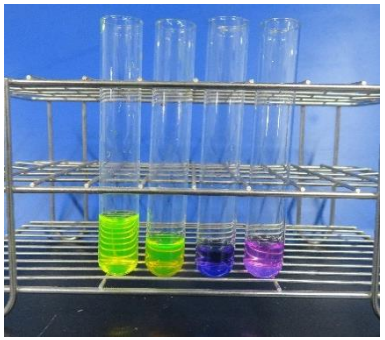
## Objective

- Learn the structures, synthesis, and the color change of the acid-base indicator phenolphthalein and its derivatives
- Synthesis of green fluorescent elastomer

## Lab techniques

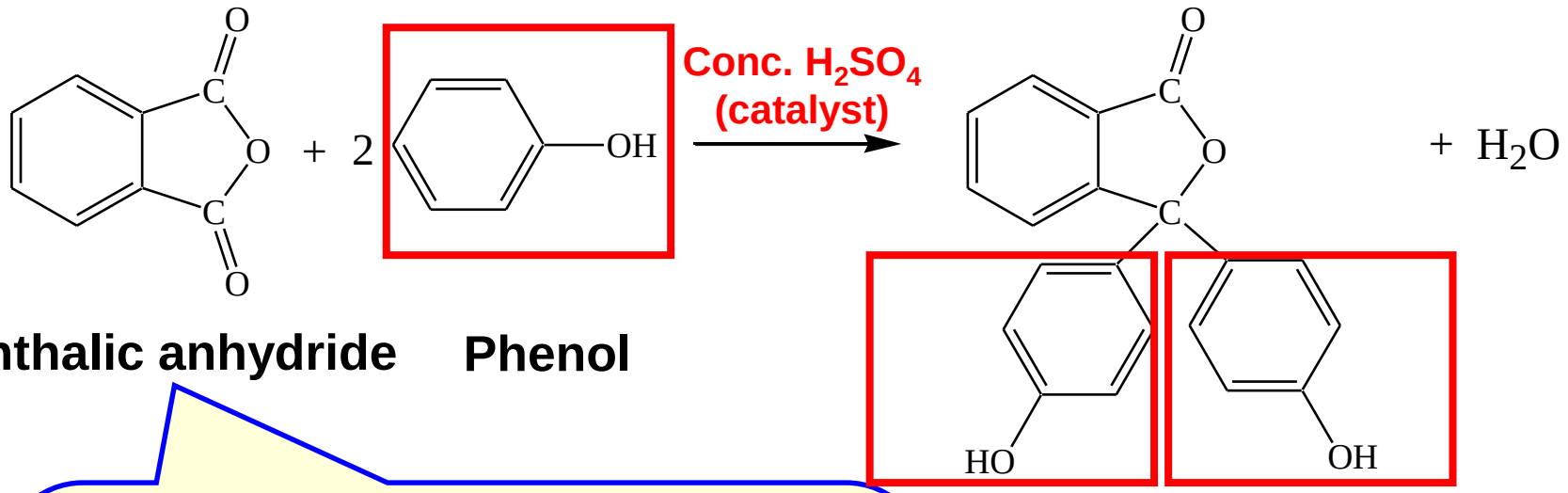
- Using the hot plate, sand bath and Vortex mixer
- Decantation



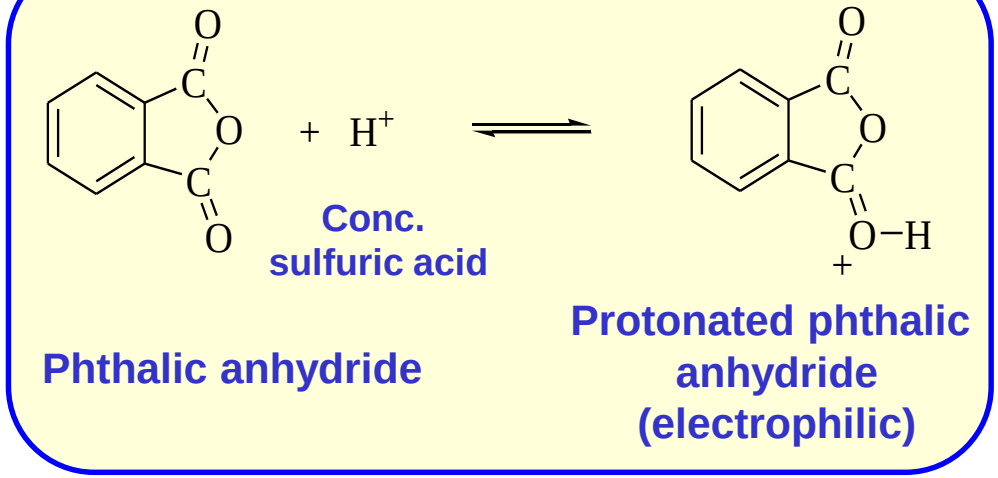
# Preparation of Phenolphthalein

I.

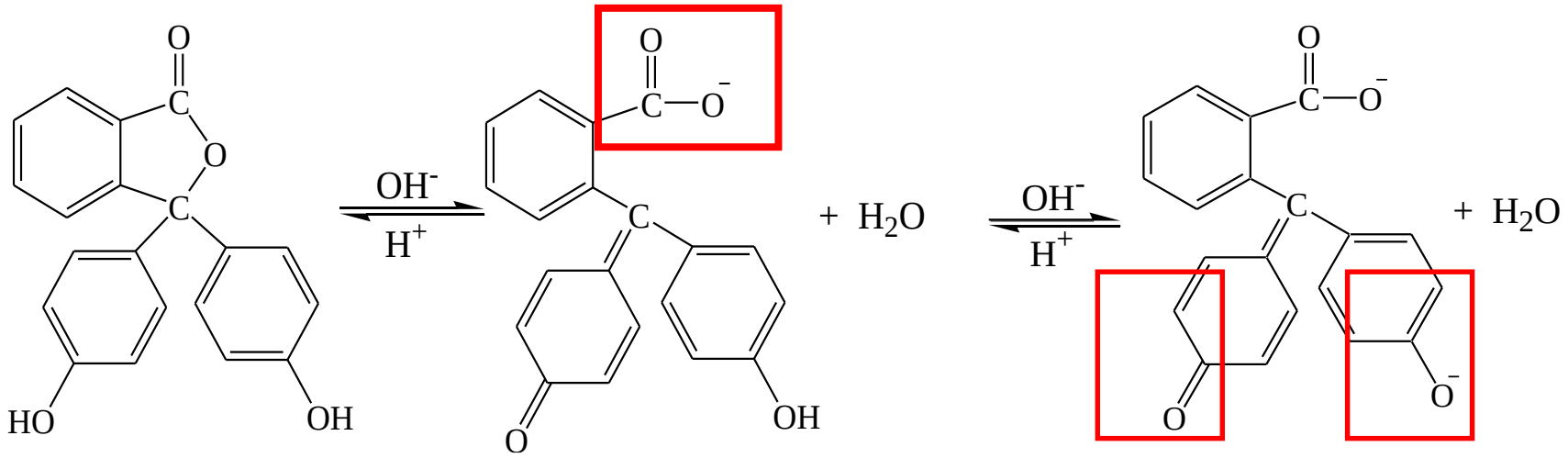



**Phthalic anhydride** **Phenol**

**Phenolphthalein**



# Color Change of Phenolphthalein



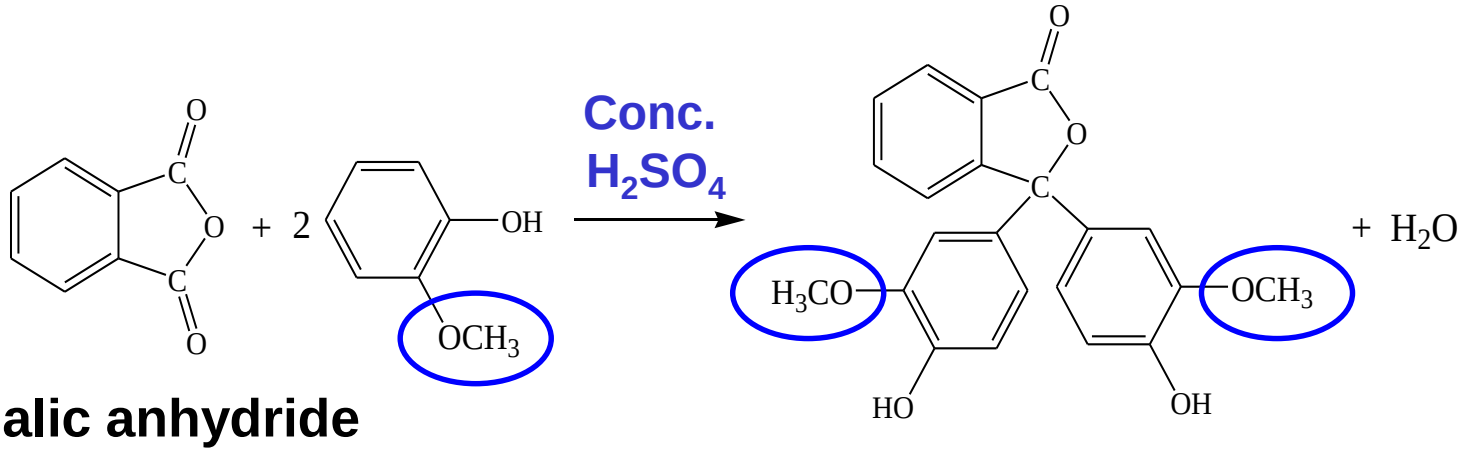

**Phenolphthalein in acidic soln (colorless)**

Phenolphthalein in neutral soln (light yellow)

**Phenolphthalein in basic soln (magenta)**


# Effect of Substituent on Color

II.

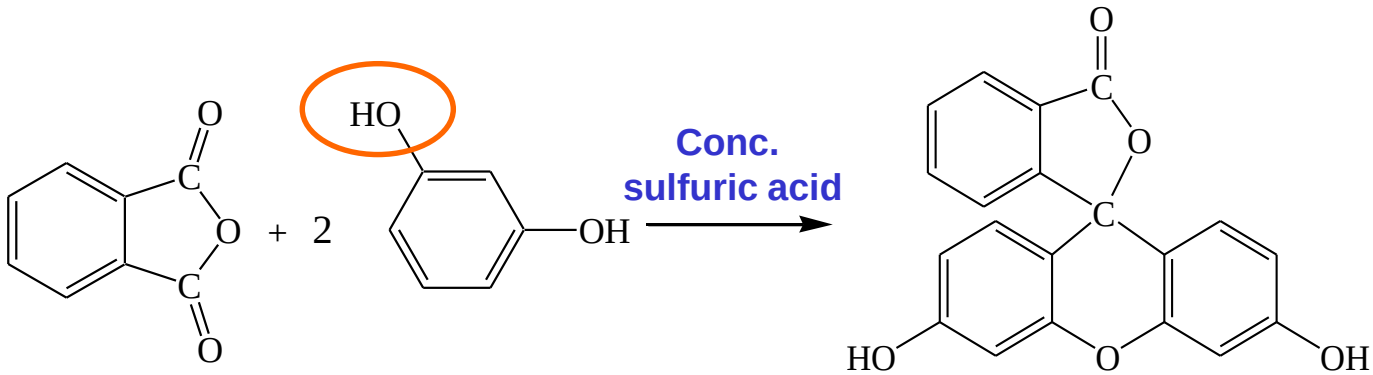



**Phthalic anhydride**

**Guaiacol**

**Guaiacolphthalein**

III.

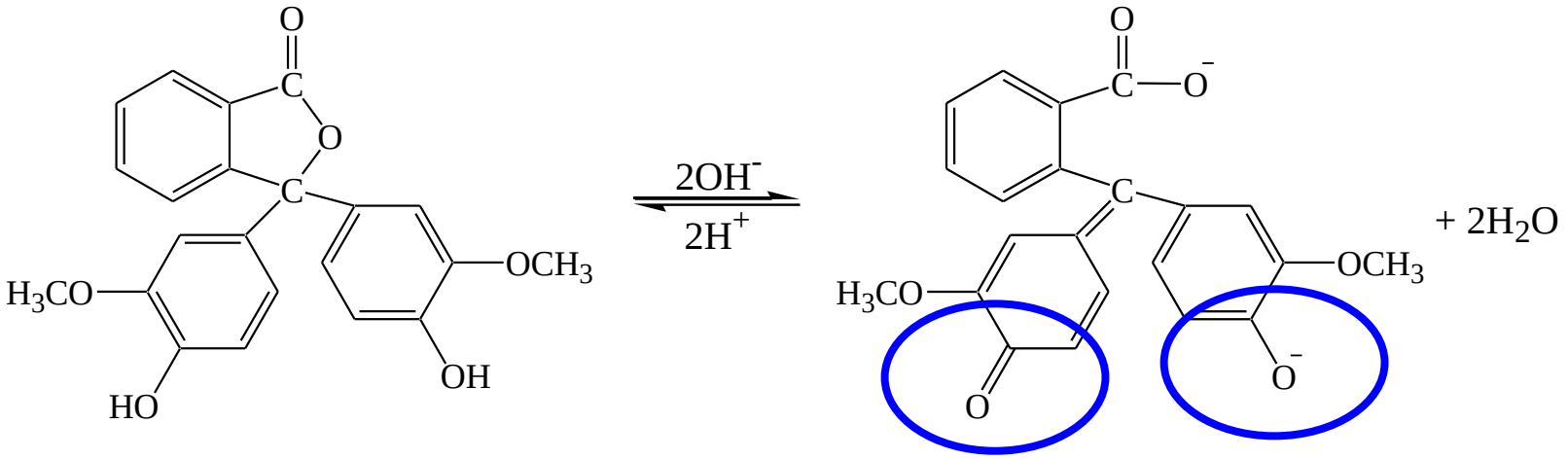



**Phthalic anhydride**

**Resorcinol**

**Fluorescein**
**(Resorcinolphthalein)**

# Color Change Of Guaiacolphthalein

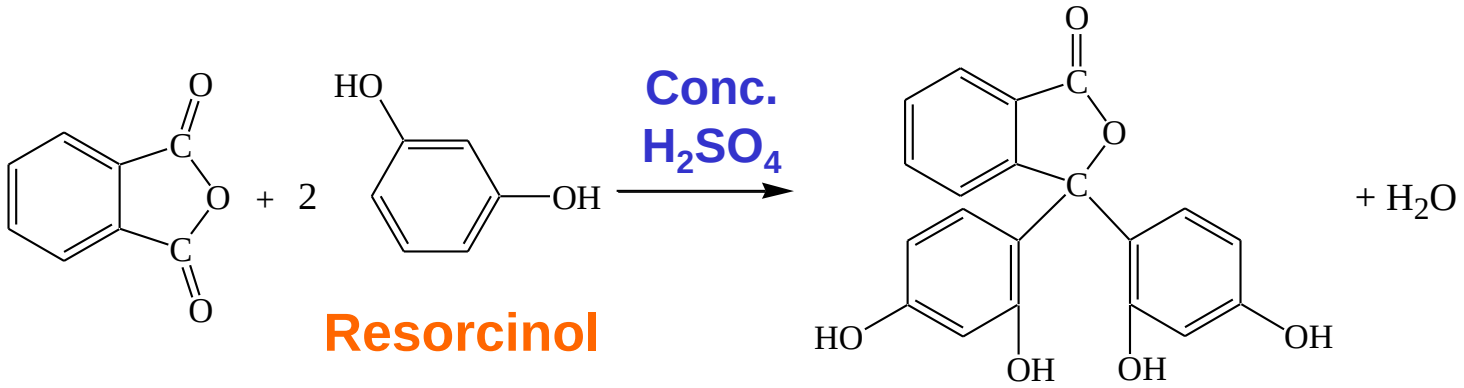



**Guaiacolphthalein in acidic soln (colorless)**

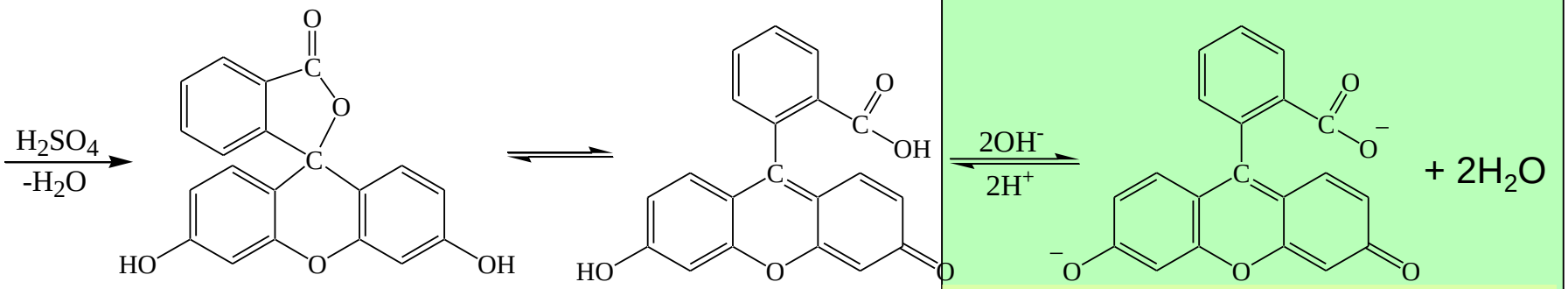
**Guaiacolphthalein in basic soln (blue)**


# Synthesis of Fluorescein

III.




**Phthalic anhydride**

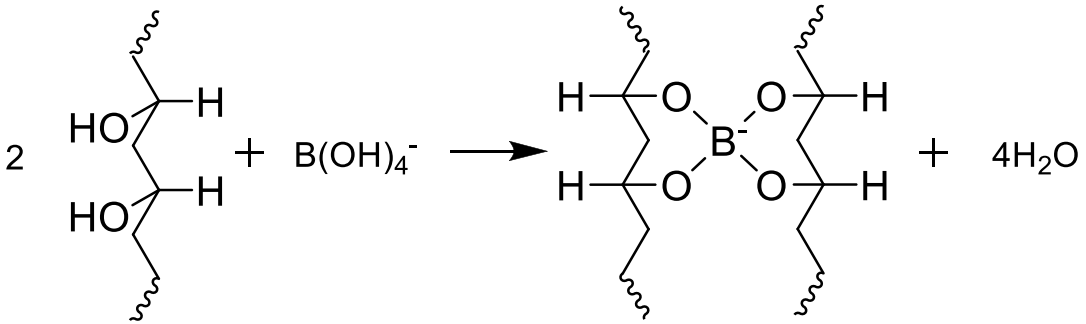



**Tautomer forms of fluorescein (Resorcinolphthalein)**

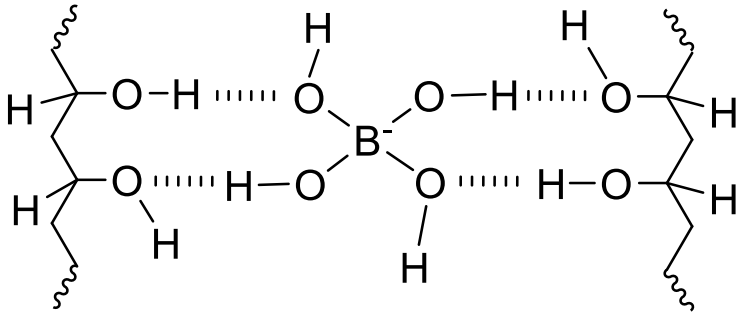
**Green fluorescing substance in basic soln**

# Fluorescent Elastomer

- 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**

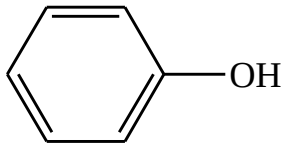
# Experiment Tasks

**Operate in fume hood**

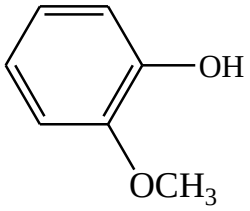
**Product**

½ spoon Phthalic anhydride

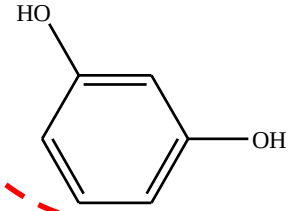
(I) 2 d Phenol




(II) 2 d Guaiacol




(III) ½ spoon Resorcinol




1 d

Conc. $H_2SO_4$

Heat and react in sand bath (150~200°C) 5-10 s

⇓

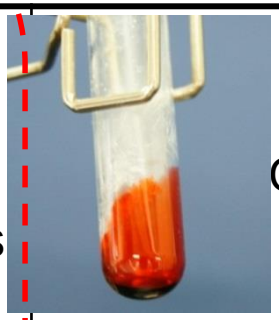
Move out and shake the tube for mixing

⇓

Repeat heating, shaking and examining the color

⇓

Stop heating when color change

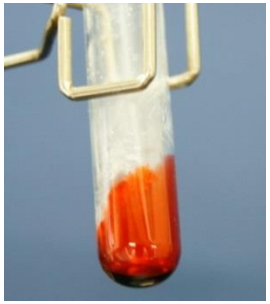
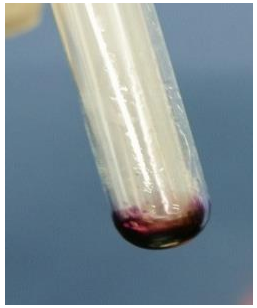


Orange

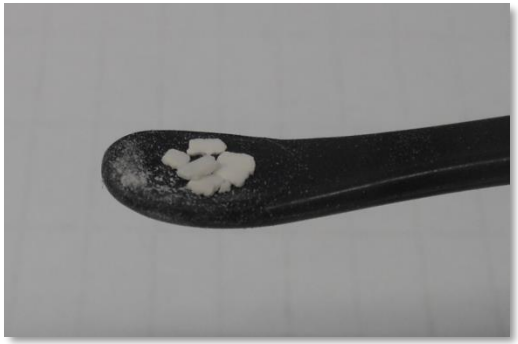
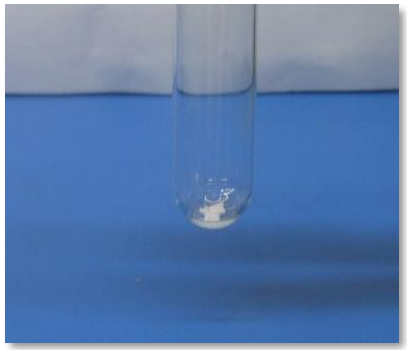
Purple

Dark brown

# Experiment Tasks

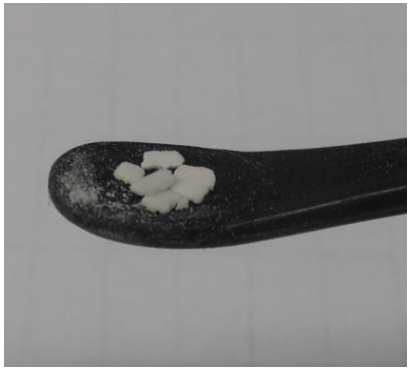
| Product from | | Operate in hood | Acid-base test | |
|---|---|---|---|---|
| (I) Phenol |  | Add 3 mL $H_2O$<br>⇓<br>Stir and mix<br>⇓<br>Product precipitate out<br>⇓<br>Decant the supernatant and get solid product | Add 1 mL 95% ethanol to dissolve the product | Add NaOH(aq)<br>⇓<br>Add HCl(aq)<br>⇓<br>Observe color change |
| (II) Guaiacol |  | | | |
| (III) Resorcinol |  | | | |

# Preparation before Synthesis

- Obtain followings in a 250 mL beaker
  - One **dry** test tube with ½ spoon phthalic anhydride
  - One test tube with 3 mL $H_2O$
  - Test tube tongs
  - Glass rod
- Wear a cotton glove outside the NBR glove for heating operation

# Step 1: Synthesis of Phenolphthalein



**Obtain dry test tube**

- Add ½ spoon of **phthalic anhydride**
- Add 2 d **phenol** and **1 d conc. sulfuric acid**

**Synthesis**

- Keep the temp. of sand bath at **150~200˚C**
- Insert the test tube in sand bath for **5-10 s**
- Move out and gently shake the test tube
- Repeat heating and mixing several times
- Stop heating till color change
- Record color

**Add 3 mL DI water**

- Stir and mix with glass rod, then product precipitate out
- Decant the supernatant
- Dissolve the solid with 1 mL 95% ethanol

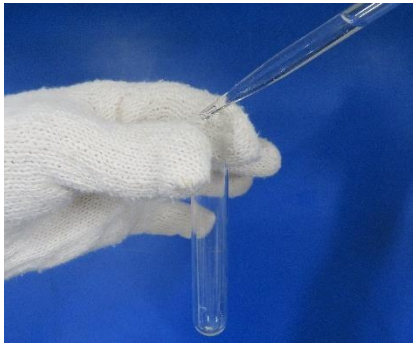
**Acid-base test**

- Take portions of soln
- Add drops of 1 *M* NaOH to observe the color change
- Add drops of 1 *M* HCl to observe the color change
- Record the color change

# Step 2: Synthesis of Guaiacolphthalein

Intermittent heating

Add DI water



Add EtOH

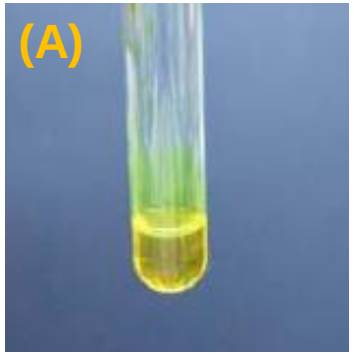

Acid-base test

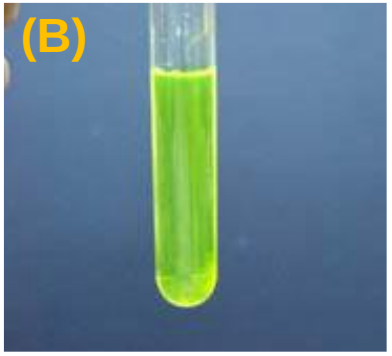
**One dry test tube**

- Add ½ spoon of phthalic anhydride
- Add 2 d **guaiacol** and 1 d conc. sulfuric acid
- Repeat heating in sand bath and mixing alternately to synthesize
- Add 3 mL of water to precipitate the product out and decant the supernatant
- Dissolve ppt in 1 mL 95% ethanol
- Examine the color change with adding NaOH(aq) and HCl(aq)

✓ Reaction is **faster** than phenol that shall avoid overheating

# Step 3.1: Synthesis of Fluorescein




**One dry test tube**

- Add ½ spoon of phthalic anhydride
- Add ½ **spoon of resorcinol** and 1 d conc. sulfuric acid
- Repeat heating and mixing to obtain fluorescein

**Obtain the product**

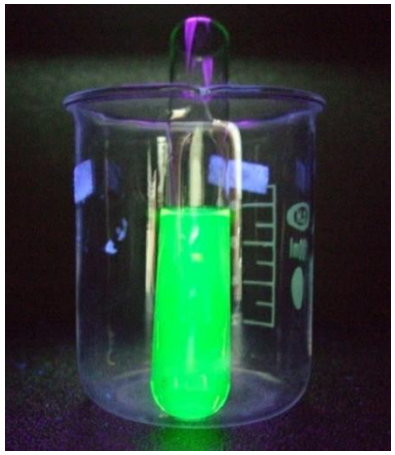
- Add 3 mL of water to precipitate the product out
- Decant the supernatant
- Take portion of product
- Dissolve with 1 mL 95% ethanol (soln A)

**Dilute the solution**

- Obtain 2~3 d of soln A into another test tube
- Dilute with 10% ethanol until light yellow (soln B)

✓ The fluorescein decomposes at 315°C
✓ Test tube should be moved in-and-out of sand bath to avoid over heating

# Step 3.2: Observe Fluorescence




**Soln B**

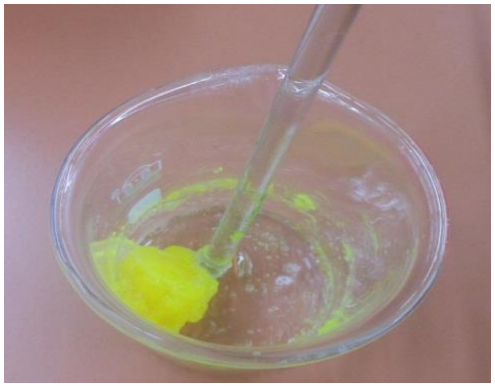
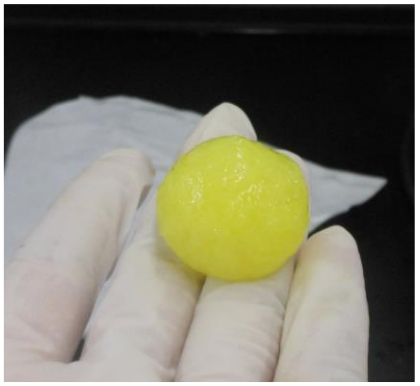
- Add drops of 1 *M* NaOH to soln B
- Observe green fluorescence under UV (examine under long / short wavelength)

**UV lamp**
**Three-stage-switch**

- Long wavelength, 366 nm
- Off
- Short wavelength, 254 nm

✓ Avoid exposing your eyes and skin under UV light

# Step 3.3: Fluorescent Elastomer

- **Highlighter DIY**
- Add 3 d fluorescein (**soln A**) and 2-3 d NaOH(aq) in 100 mL beaker
- Add PVA glue to make a thin film that covers the bottom of beaker
- Mix thoroughly with glass rod and record the color change
- Use a cotton bud to absorb some sticky glue and write on paper
- **Fluorescent elastomer**
- Add sodium tetraborate solution drop by drop to the above sticky glue
- Mix thoroughly with glass rod
- Observe the change in viscosity

# Additional Notes

- Phenol, conc. sulfuric acid…etc. are **corrosive**; wear NBR gloves and avoid contacting with skin and eyes
- The amount of chemicals used in this experiment does not need to be precise
- Take little amount of reactants to prevent chemical waste which are corrosive and volatile
- **Operate heating in the fume hood and avoid burns**
- Heat the reactants for *ca.* 5-10 s., and move out of sand bath alternately to avoid overheating
- **Prevent exposing eyes and skin from UV light**

# Clean-Up and Check-Out

- **Dissolve the waste product with 10% alcohol**, pour the first rinsed waste liquid into recycling bin then clean with water
- Brush and wash the test tubes thoroughly
- Wash hands after experiment
- Wash the lab coat alone

- Clean up the lab bench and check personal equipment inventory (have an associate TA sign the check list)
- This is a **Brief Report** experiment:
  - **Hand in prelab/lab note/report together to the TA**

- Groups on duty shall stay and help clean up the lab