Synthesis of Acid-Base Indicators

- Wash clean 3 test tubes and hand 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

(2020/05/08 revised)
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.

\[
\text{Phthalic anhydride} + 2 \text{ Pheno} \xrightarrow{\text{Conc. H}_2\text{SO}_4 \text{ (catalyst)}} \text{Phenolphthalein} + \text{H}_2\text{O}
\]

Phthalic anhydride

[Pheno]

Conc. sulfuric acid

Protonated phthalic anhydride (electrophilic)
Color of Phenolphthalein Indicator

Phenolphthalein in acidic soln (colorless)  Phenolphthalein in neutral soln (light yellow)  Phenolphthalein in basic soln (magenta)
Effect of Substituent on Color

II.

\[ \text{Phthalic anhydride} + 2 \text{Guaiacol} \xrightarrow{\text{Conc. } \text{H}_2\text{SO}_4} \text{Diguaiacol phthalein} + \text{H}_2\text{O} \]

III.

\[ \text{Phthalic anhydride} + 2 \text{Resorcinol} \xrightarrow{\text{Conc. sulfuric acid}} \text{Fluorescent Yellow} \]
II. 

Diguaiacol phthalein in acidic soln (colorless)

\[
\text{Diguaiacol phthalein in basic soln (blue)}
\]

\[
\text{Diguaiacol phthalein in acidic soln (colorless)}
\]

\[
\begin{align*}
\text{Diguaiacol phthalein in basic soln (blue)}
\end{align*}
\]
Synthesis of Fluorescent Yellow

III.

Phthalic anhydride + Resorcinol

\[
\text{Phthalic anhydride} + 2\text{Resorcinol} \xrightarrow{\text{Conc. } \mathrm{H}_2\mathrm{SO}_4} \text{Fluorescent Yellow}
\]

Green fluorescing substance in basic soln

Tautomer

\[
\text{Fluorescent Yellow} \leftrightarrow \text{Tautomer}
\]

\[\text{Fluorescent Yellow} + 2\text{OH}^- \xrightarrow{2\text{H}^+} \text{Green fluorescing substance in basic soln} + 2\text{H}_2\text{O}\]
Flow Chart I

<table>
<thead>
<tr>
<th>Operate in hood</th>
<th>React</th>
<th>Product</th>
</tr>
</thead>
</table>
| ½ spoon Phthalic anhydride | Stir and mix  
  Heat on flame  
  React  
  Color change  
  Solid disappear | Orange  
 Purple  
 Dark brown |
| 2 d phenol | 2 d Conc. H₂SO₄ | 2 d guaiacol  
  ½ spoon resorcinol |
### Flow Chart II

<table>
<thead>
<tr>
<th>Product from</th>
<th>Add H₂O in hood</th>
<th>Acid-base test</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Phenol</strong></td>
<td>Add 3 mL H₂O</td>
<td>Add NaOH(aq)</td>
</tr>
<tr>
<td></td>
<td>⇓</td>
<td>⇓</td>
</tr>
<tr>
<td></td>
<td>Stir and mix</td>
<td>Add HCl(aq)</td>
</tr>
<tr>
<td></td>
<td>⇓</td>
<td>⇓</td>
</tr>
<tr>
<td></td>
<td>Product</td>
<td>Observe</td>
</tr>
<tr>
<td></td>
<td>precipitate out</td>
<td>color</td>
</tr>
<tr>
<td></td>
<td>⇓</td>
<td>change</td>
</tr>
<tr>
<td></td>
<td>Decant</td>
<td></td>
</tr>
<tr>
<td></td>
<td>supernatant</td>
<td></td>
</tr>
<tr>
<td></td>
<td>and get solid</td>
<td></td>
</tr>
<tr>
<td></td>
<td>product</td>
<td></td>
</tr>
</tbody>
</table>

- Flow Chart II is a diagram illustrating the process of handling phenol, guaiacol, and resorcinol.
- Phenol is treated by adding 3 mL of H₂O, stirring and mixing, and obtaining a solid product.
- Guaiacol involves adding H₂O in a hood, followed by the addition of NaOH(aq) and observing a color change.
- Resorcinol sees H₂O added in a hood, leading to the addition of HCl(aq) and observing a color change after dissolving in 95% ethanol.
Step 1  Preparation

One 250 mL beaker
1. One dry test tube with ½ spoon phthalic anhydride
2. A test tube with 3 mL H₂O
3. Glass rod
4. Test tube holder
Step 2  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

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 reversibly

- Phenol and sulfuric acid are corrosive.
- Wear gloves
Step 3  Synthesis of Diguaiacol Phthalein

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

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
Step 4  Synthesis of Fluorescent Yellow

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

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
Step 5  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.

\[ 2 \text{HO-CH}_2\text{-OH} + \text{B(OH)}_4^- \rightarrow \text{HO-CH}_2\text{B(OH)}_2\text{-CH}_2\text{OH} + 4\text{H}_2\text{O} \]

Cross-linking with covalent bonding  Cross-linking with hydrogen-bonding
Step 6 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 borate soln. drop by drop
- 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, heat the reactants for ca. 5 s., and move 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
T1 - Alcohol Lamp

- Inspect the lamp before each use to make sure there are no cracks, chips or defects in the glass body.
- Adjust the wick height to about 3 mm from the top of the metal cap.
- Fill the lamp with denatured or 95% ethanol through a suitable funnel to about ½ to 2/3 volume.
- Light the alcohol lamp using a match.
- Use the lamp only in an upright position.
- Keep all combustible materials (clothing, paper, books, chemicals, etc.) away from the lamp when in use.
- Never use book or other items to raise the alcohol lamp.
- Use windshield to block wind and do not use books. Do not tilt lamp to avoid alcohol leakage while heating.
- Adjust device upon heating to an appropriate height.
- After use, cap should be put on to insulate air and extinguish the flame instead of blow it out. If lamp will not be used for a long time, alcohol inside should be returned to storage bottle.
- If lamp is overturned and cause small fire, cover the fire quickly with a wet rag and do not remove the rag immediately while extinguished, or flame may ignite again. Use fire extinguisher instead if a big fire happens. Inform teacher as soon as possible in such emergency and after fire is extinguished, immediately open doors, windows and exhausts to expel alcohol vapor in lab.