

Chapter: 05

Ch.Id:-ASU/GRF/EB/AEHFPOC/2022/Ch-05

DOI: <https://doi.org/10.52458/9789391842697.2022.eb.grf.asu.ch-05>

AIM: TO PERFORM SYSTEMATIC QUALITATIVE ANALYSIS OF PHENOLS

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Requirements

Chemicals

1. Glacial acetic acid
2. Bromine water
3. FeCl_3
4. Phenol
5. Sodium nitrite
6. H_2SO_4
7. NaOH
8. Phthalic acid
9. dil. HCl
10. Aniline
11. Benzoyl chloride

Glass wares

1. Test-tube
2. Stands
3. Brush
4. Holder
5. Glass rod
6. Beakers

Theory: The term "phenol" refers to either the hydroxyl group (-OH) that is attached to an aromatic ring or, more generally, to the hydroxy derivatives of aromatic compounds. Phenols are acids,

however they are much weaker than carboxylic acids. It is simple to go through the substitution reaction. Phenol is well recognized as one of the most significant and versatile organic chemicals used in industry.

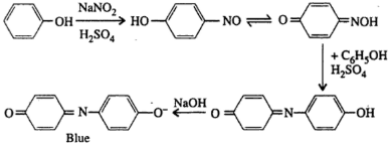
Reagents Preparation

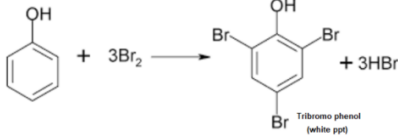
- **Ferric chloride solution:** A neutral FeCl_3 solution is made by adding dil. NaOH solution drop by drop to a FeCl_3 solution until a small but permanent brown precipitate forms. Filter the solution and utilise the filtrate for the experiment.
- **Bromine Water:** Add 5 ml Br_2 to 100 ml of water and shake vigorously. Discard the transparent liquid.

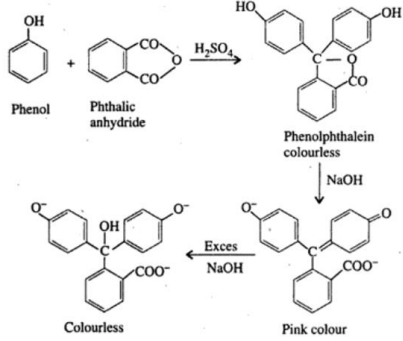
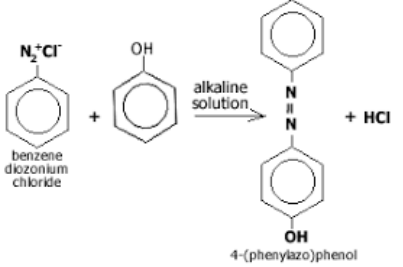
Functional Group Test for Phenols

S. No.	Identification Test	Observation	Inference
1	Litmus Test: Place a drop of the given organic solution or a tiny crystal on a piece of moist litmus paper. Change in colour is observed; if it turns red, it indicates presence of acidic groups that may be phenolic group. Likewise, carboxylic acid passes this test. Compared to carboxylic acid, phenol is mildly acidic and does not react with aqueous NaHCO_3 to release CO_2 bubbles.	Blue litmus paper turns red	Acidic group may be present
2	Ferric Chloride Test: Given organic sample is dissolved in H_2O . Drop by drop, add a	A red, blue, green or purple	Acidic group may be

	<p>neutral FeCl_3 solution. Observe the colour shift.</p> <p>A coloured complex is produced when an aqueous solution of phenol combines with a freshly prepared solution of FeCl_3. Most phenols produce solutions with a dark hue. The chemical reaction is described in the following section.</p> $6\text{C}_6\text{H}_5\text{OH} + \text{FeCl}_3 \longrightarrow [\text{Fe}(\text{C}_6\text{H}_5\text{O})_6]^{3-} + 3\text{HCl} + 3\text{H}^+$ <p style="text-align: center;">(violet colour complex)</p>	<p>colouration</p>	<p>present</p>
<p>3</p>	<p>Liebermann's Test: Sodium nitrite crystal put in a clean and dry test tube. Then, mix 1 ml of phenol in a solution of sodium nitrite. Allow the mixture to cool after being heated gently. Then, add 1 ml of concentrated H_2SO_4 and shake the mixture. Observe the solution's colour shift. If the supplied chemical has a phenolic group, dilute the solution until the compound becomes red. After adding NaOH solution, a blue or green solution emerges.</p> <p>The reaction between phenol, conc. H_2SO_4, and sodium nitrite produces a yellow Quinone monoxime complex. With an excess of phenol and H_2SO_4, a</p>	<p>Blue or green colour solution</p>	<p>Phenol is confirmed</p>

	<p>complex of indophenol with a deep blue colour is formed. On dilution, red-colored indophenol is produced, which, when treated with NaOH, transforms into a deep blue-colored sodium salt solution of indophenol.</p> <p>Note: This test is given by phenols which contain a free para position.</p> 		
<p>4</p>	<p>Bromine Water Test: In a clean and dry test tube, sample is dissolved in glacial acetic acid. Br₂ water solution is added drop by drop to the test solution.</p> <p>Br₂ and phenol undergo an electrophilic substitution reaction. When Br₂ water is added to a phenol aqueous solution, the brown colour of the bromine vanishes and a white precipitate of tribromophenol forms. The chemical reaction is described in the following section.</p>	<p>Colour of bromine disappears</p>	<p>Phenol is confirmed</p>

			
<p>5</p>	<p>Phthalein Dye Test: In a test tube, place the organic component to be evaluated. Add 200mg of phthalic anhydride. Add concentrated H₂SO₄ to the mixture. Warm the solution for a couple of minutes. Pour the cooled mixture into a beaker containing diluted NaOH solution. Dilute the entire mixture with water in equal volume. Observe the colour change in a white background.</p> <p>Phenol, when heated with phthalic anhydride in the presence of concentrated H₂SO₄, produces phenolphthalein, a colourless condensation product. On subsequent interaction with diluted NaOH solution, the pink fluorescent chemical fluorescein is produced. Different phenolic compounds give distinctive hues that are visible on a white background. The chemical reaction is described in the following section.</p>	<p>Presence of fluorescence colour (view in black background)</p>	<p>Phenol is confirmed</p>

	 <p>Phenol + Phthalic anhydride $\xrightarrow{H_2SO_4}$ Phenolphthalein (colourless)</p> <p>Phenolphthalein (colourless) \xrightarrow{NaOH} Pink colour</p> <p>Pink colour $\xrightarrow{Excess NaOH}$ Colourless</p>		
<p>6</p>	<p>Azo dye test: Dissolve two drops of aniline in one millilitre of diluted HCl, cool on ice, and add a saturated sodium nitrite solution. Now, it is added to a well-chilled sample solution (phenols) in aqueous NaOH.</p> <p>A red colourant is produced. Aryldiazonium salts react with aromatic phenol rings to produce brightly coloured azo compounds (dye). These are referred to as coupling reactions.</p>  <p>benzene diazonium chloride + phenol $\xrightarrow{\text{alkaline solution}}$ 4-(phenylazo)phenol + HCl</p>	<p>Red coloured dye</p>	<p>Phenol is confirmed</p>

7	Benzoylation test: Dissolve 0.1 g of the material in 5 millilitres of 20% NaOH. Mix the above test solution with 1 mL of benzoyl chloride and vigorously mix with care for 15 minutes. $\text{Ar-OH} + \text{C}_6\text{H}_5\text{COCl} \longrightarrow \text{C}_6\text{H}_5\text{COOAr} + \text{HCl}$	White ppt	Phenol is confirmed
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Result: The results of the systemic qualitative tests performed and phenol was found and reported.