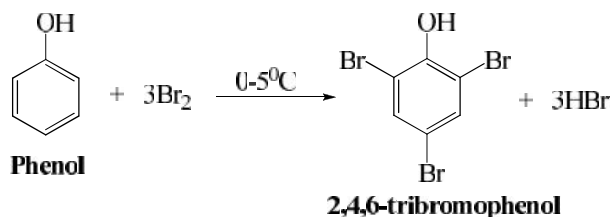


**Aim** : To prepare 2,4,6-tribromophenol from phenol and determine its practical yield, theoretical yield and percentage of yield. (Bromination - Nitration – Aromatic Electrophillic Substitution Reaction) (right side)

**Reaction** : (left side)



**Requirements** : (right side)

**Glasswares** - Conical flask, test tube, cotton, beaker of 50/100 ml, Funnel, Glassrod, ice bath

**Chemicals** - 1 gm phenol, 2 mL liquid bromine, sodium bisulphate solution if necessary

**Procedure** : (right side)

Take 1 gm phenol in a beaker and dissolve it in 50 mL water. Keep the flask in an ice bath for 10 minutes. Now take 2 mL liquid bromine in a test tube and plug the test tube with cotton immediately. Keep this test tube in an ice bath for 5 minutes. Dropwise add this cooled liquid bromine from test tube to cold solution of phenol with vigorous shaking. Yellow coloured solid will separate out which is 2,4,6-tribromophenol. [If the colour of solution is dark yellow orange, then add small amount of sodium bisulphite solution to decolourize it]. Filter this solid, wash with cold water, dry it and weigh. Calculate practical yield, theoretical yield and percentage of yield. Recrystallize from R-spirit and take its melting point. (M.P. is 95-96 °C).

**Calculation** : (left side)

(1) **Practical Yield:** \_\_\_\_\_ gms (A gm)

(2) **Theoretical yield :**

$$\begin{aligned}
 94 \text{ gm Phenol} &= 331 \text{ gm 2,4,6-tribromophenol} \\
 X \text{ gm Phenol} &= \frac{X \times 331}{94} = \text{_____ gm (B gm)}
 \end{aligned}$$

(3) **Percentage of yield :**

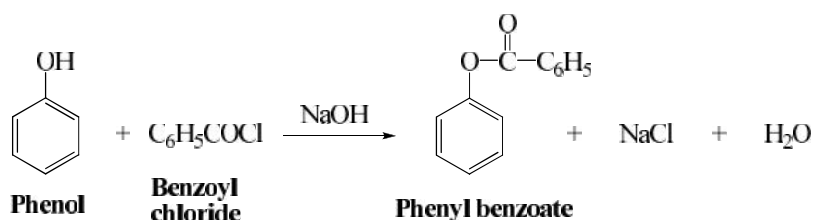
$$\begin{aligned}
 \text{If yield is B gm} &= \% \text{ of yield is } 100 \\
 A \text{ gm} &= \frac{100 \times A}{B} = \text{_____ \%}
 \end{aligned}$$

**Result** : (right side)

Structure of the product	MP °C	Practical Yield (gm)	Theoretical Yield (gm)	Percentage of Yield (%)

**Aim :** To prepare *phenyl benzoate* from *phenol* and determine its practical yield, theoretical yield and percentage of yield. (right side)

**Reaction :** (left side)



**Requirements :** (right side)

**Glasswares** - stoppered bottle, Test tube, Beaker of 50/100 ml, Funnel, Glassrod, tripod stand

**Chemicals** - 1 gm phenol, 2 mL benzoyl chloride, 12 mL 10% NaOH solution

**Procedure :** (right side)

Take 1 gm phenol in a stoppered bottle containing 12 mL 10% NaOH. Now slowly and gradually add 2 mL benzoyl chloride taken in a test tube. Grease the stopper of the reaction bottle lightly with Vaseline and cap it on the bottle, then, holding the stopper in place securely, shake the bottle vigorously at intervals for a period of 15 minutes. After some time white ppt of phenyl benzoate will separate out and simultaneously the unpleasant smell of benzoylchloride disappears.

Add 10 mL of distill water to this alkaline mixture and shake well. Filter this solid, wash with water, dry it and weigh. Calculate practical yield, theoretical yield and percentage of yield. Recrystallize from R-spirit and take its melting point. (M.P. is 68-69 °C).

**Calculation :** (left side)

(1) **Practical Yield:** \_\_\_\_\_ gms (A gm)

(2) **Theoretical yield :**

$$\begin{aligned} 94 \text{ gm Phenol} &= 198 \text{ gm phenyl benzoate} \\ X \text{ gm Phenol} &= \frac{X \times 198}{94} = \text{_____ gm (B gm)} \end{aligned}$$

(3) **Percentage of yield :**

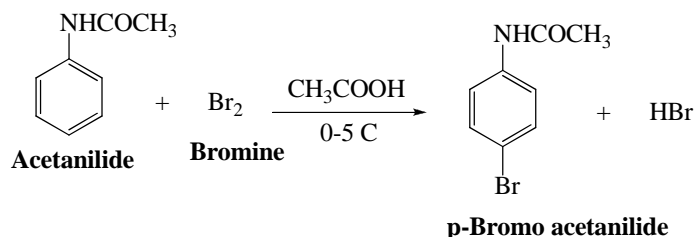
$$\begin{aligned} \text{If yield is B gm} &= \% \text{ of yield is } 100 \\ A \text{ gm} &= \frac{100 \times A}{B} = \text{_____ \%} \end{aligned}$$

**Result :** (right side)

Structure of the product	MP °C	Practical Yield (gm)	Theoretical Yield (gm)	Percentage of Yield (%)

**Aim :** To prepare *p-bromoacetanilide* from *acetanilide* and determine its practical yield, theoretical yield and percentage of yield. (Bromination - Nitration – Aromatic Electrophilic Substitution Reaction) (right side)

**Reaction :** (left side)



**Requirements :** (right side)

**Glasswares** - Test tube, two beakers of 50/100 ml, Funnel, Glassrod

**Chemicals** - 1 gm acetanilide, 3.5 mL glacial acetic acid, 2.5 mL bromine in glia. Acetic acid

**Procedure :** (right side)

Take 1 gm acetanilide in a beaker and dissolve it in 3.5 mL glacial acetic acid. Keep the beaker in an ice bath for 10 minutes. Now dropwise add 2.5 mL solution of bromine in acetic acid taken in test tube and stir well. The color of mixture turns orange due to bromination reaction. Keep the reaction mixture at room temperature for half an hour and then pour it in a beaker containing some crushed ice and water. *p*-Bromo acetanilide separates out on stirring. Filter this solid, wash with cold water, dry it and weigh. Calculate practical yield, theoretical yield and percentage of yield. Recrystallize from R-spirit and note down its melting point. (M.P. is 166-167 °C).

**Calculation :** (left side)

(1) **Practical Yield:** \_\_\_\_\_ gms (A gm)

(2) **Theoretical yield :**

$$135 \text{ gm acetanilide} = 214 \text{ gm } p\text{-bromo acetanilide}$$

$$X \text{ gm acetanilide} = \frac{X \times 214}{135} = \text{_____ gm (B gm)}$$

(3) **Percentage of yield :**

$$\text{If yield is B gm} = \% \text{ of yield is } 100$$

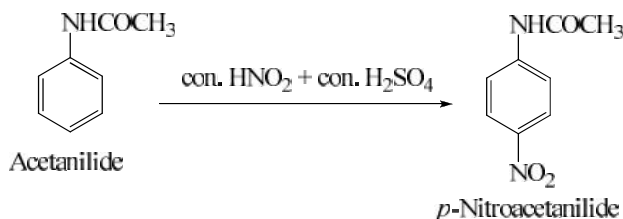
$$A \text{ gm} = \frac{100 \times A}{B} = \text{_____ \%}$$

**Result :** (right side)

Structure of the product	MP °C	Practical Yield (gm)	Theoretical Yield (gm)	Percentage of Yield (%)

**Aim :** To prepare *p*-nitroacetanilide from acetanilide and determine its practical yield, theoretical yield and percentage of yield. (Nitration – Aromatic Electrophilic Substitution Reaction) (right side)

**Reaction :** (left side)



**Requirements :** (right side)

**Glasswares** - Two beakers of 50/100 ml, Funnel, Glassrod, ice bath

**Chemicals** - 1 gm acetanilide, 1 mL glacial acetic acid, 2 mL con. H<sub>2</sub>SO<sub>4</sub>, nitrating mixture (0.5 mL con. HNO<sub>3</sub> + 0.5 mL con. H<sub>2</sub>SO<sub>4</sub>)

**Procedure :** (right side)

Take 1 gm acetanilide in a beaker and add 1 mL glacial acetic acid and stir well. Now add 2 mL of con. H<sub>2</sub>SO<sub>4</sub> and shake well until clear solution is obtained. Keep this beaker in a freezing bath (ice + salt) for about 10 minutes. Take 1 ml nitrating mixture in a test tube and dropwise add it to it to the above solution with stirring (temperature of the solution should not exceed 10 °C while nitrating mixture is being added). Keep this mixture at room temperature for 30 min. and then pour the mixture in an ice cold water, white crystals of *p*-nitroacetanilide separates out. Filter this solid, wash with cold water, dry it and weigh. Calculate practical yield, theoretical yield and percentage of yield. Recrystallize from R-spirit and note down its melting point. (M.P. is 214-215 °C).

**Calculation :** (left side)

(1) **Practical Yield:** \_\_\_\_\_ gms (A gm)

(2) **Theoretical yield :**

$$\begin{aligned}
 135 \text{ gm acetanilide} &= 180 \text{ gm } p\text{-nitro acetanilide} \\
 X \text{ gm } p\text{-nitro acetanilide} &= \frac{X \times 180}{135} = \text{_____ gm (B gm)}
 \end{aligned}$$

(3) **Percentage of yield :**

$$\begin{aligned}
 \text{If yield is B gm} &= \% \text{ of yield is } 100 \\
 A \text{ gm} &= \frac{100 \times A}{B} = \text{_____ \%}
 \end{aligned}$$

**Result :** (right side)

Structure of the product	MP °C	Practical Yield (gm)	Theoretical Yield (gm)	Percentage of Yield (%)