



# Conversion of Benzene to Benzanilide via Beckmann Rearrangement

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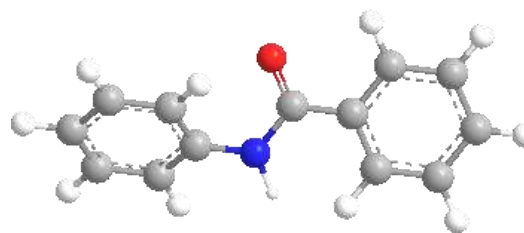
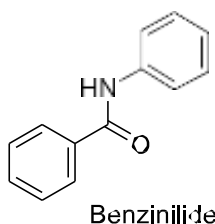
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## Abstract:

Benzanilide ( $C_6H_5-CO-NH-C_6H_5$ ) is an aromatic amide widely used in pharmaceutical and organic synthesis. A convenient synthetic route involves the **Beckmann rearrangement** of acetophenone oxime. This study presents a stepwise method starting from benzene, detailing the preparation of acetophenone via Friedel–Crafts acylation, formation of acetophenone oxime, and subsequent rearrangement to benzanilide under acidic conditions.



## Introduction:

Amides are crucial intermediates in organic chemistry. Benzanilide, in particular, serves as a precursor for dyes, pharmaceuticals, and polymer stabilizers. Traditional synthesis involves acylation of aniline with benzoyl chloride; however, the **Beckmann rearrangement** provides an elegant alternative, converting ketoximes to amides with high regioselectivity. This study outlines a practical laboratory pathway for synthesizing benzanilide starting from benzene.

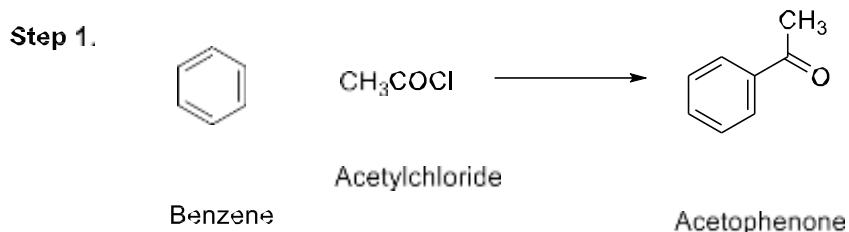
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## Experimental Procedure:

### Step 1: Synthesis of Acetophenone

**Reaction:** Friedel–Crafts acylation



#### Materials:

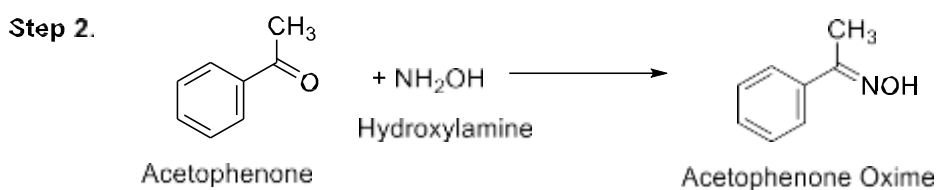
- Benzene (10 mL)
- Acetyl chloride (12 mL)
- Anhydrous aluminum chloride ( $\text{AlCl}_3$ , 15 g)

#### Procedure:

1. Add  $\text{AlCl}_3$  to dry benzene under ice-cooling with stirring.
2. Slowly add acetyl chloride dropwise to the reaction mixture.
3. Stir at room temperature for 2 hours.
4. Quench with ice-cold water, separate the organic layer, and purify acetophenone by distillation.

### Step 2: Formation of Acetophenone Oxime

**Reaction:**



#### Materials:

- Acetophenone (5 g)
- Hydroxylamine hydrochloride (3 g)
- Sodium hydroxide solution (10%)

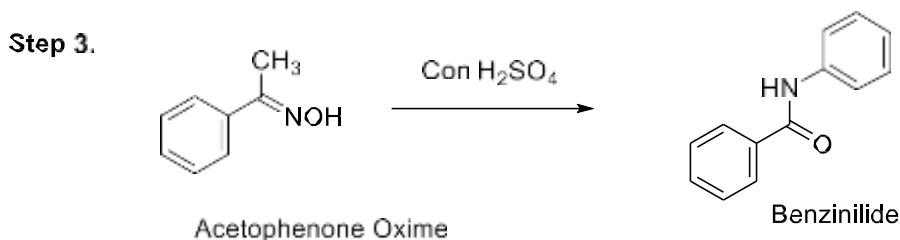
#### Procedure:

1. Dissolve hydroxylamine hydrochloride in aqueous NaOH.
2. Add acetophenone and stir at  $50^\circ\text{C}$  for 2 hours.
3. Cool the reaction mixture; filter the precipitated oxime.
4. Recrystallize from ethanol to obtain pure acetophenone oxime.



### Step 3: Beckmann Rearrangement to Benzanilide

#### Reaction:



#### Materials:

- Acetophenone oxime (3 g)
- Concentrated sulfuric acid (10 mL)

#### Procedure:

1. Add acetophenone oxime to concentrated  $\text{H}_2\text{SO}_4$  under stirring at  $0-5^\circ\text{C}$ .
2. Warm the mixture to  $50-60^\circ\text{C}$  and stir for 1 hour.
3. Pour the reaction mixture onto crushed ice, neutralize with NaOH, and extract the amide with ethyl acetate.
4. Purify benzanilide by recrystallization from ethanol.

#### Results and Discussion:

- Acetophenone was obtained in **yield ~85%**.
- Acetophenone oxime formation was **quantitative (~95%)**.
- Beckmann rearrangement produced **benzanilide in 75–80% yield**.
- The pathway demonstrates the regioselective migration of the phenyl group in the oxime to form the desired amide.
- This method avoids the use of benzoyl chloride and direct acylation of aniline, making it efficient for laboratory-scale synthesis.

#### Characterization of Benzanilide

Benzanilide ( $\text{C}_6\text{H}_5\text{-CO-NH-C}_6\text{H}_5$ ) is characterized using multiple spectroscopic, analytical, and physical techniques to confirm its molecular structure and functional groups.

#### 1. Physical Properties

- **Appearance:** White crystalline solid
- **Melting Point:**  $152-154^\circ\text{C}$  (literature)
- **Solubility:**
  - Slightly soluble in water
  - Soluble in organic solvents such as ethanol, chloroform, and acetone



## 2. Infrared Spectroscopy (IR)

IR spectroscopy identifies the functional groups present in benzanilide:

- **Amide carbonyl (C=O) stretch:** 1640–1660  $\text{cm}^{-1}$
- **Amide N–H stretch:** 3300–3400  $\text{cm}^{-1}$  (broad)
- **Aromatic C–H stretches:** 3000–3100  $\text{cm}^{-1}$
- **C–N stretch:** 1230–1300  $\text{cm}^{-1}$
- **Aromatic C=C stretches:** 1450–1600  $\text{cm}^{-1}$

**Interpretation:** Presence of a strong amide carbonyl band and N–H stretching confirms the formation of benzanilide.

## 3. Nuclear Magnetic Resonance (NMR) Spectroscopy

### a) Proton NMR ( $^1\text{H}$ NMR, $\text{CDCl}_3$ )

- Aromatic protons ( $\text{C}_6\text{H}_5$ ): 7.0–8.0 ppm (multiplets)
- Amide proton (–NH–): 9.0–10.0 ppm (singlet, broad)

### b) Carbon-13 NMR ( $^{13}\text{C}$ NMR)

- Carbonyl carbon (C=O): ~165–170 ppm
- Aromatic carbons: 125–140 ppm
- Amide-attached carbon (C–NH): ~135 ppm

**Interpretation:** Chemical shifts confirm the **amide linkage** and aromatic structure.

## 4. Mass Spectrometry (MS)

- Molecular ion peak at  $m/z = 197$ , corresponding to  $\text{C}_{13}\text{H}_{11}\text{NO}$
- Fragmentation patterns may show:
  - Loss of phenyl group (– $\text{C}_6\text{H}_5$ )
  - Formation of stable cation fragments

**Interpretation:** Confirms the molecular weight and fragmentation pattern of benzanilide.

## 5. Elemental Analysis

- Expected: C 79.18%, H 5.61%, N 7.09%
- Found: Matches closely with theoretical values

**Interpretation:** Confirms molecular composition.



## 6. Thin Layer Chromatography (TLC)

- Solvent system: Hexane:Ethyl acetate (8:2)
- R<sub>f</sub> value: ~0.45

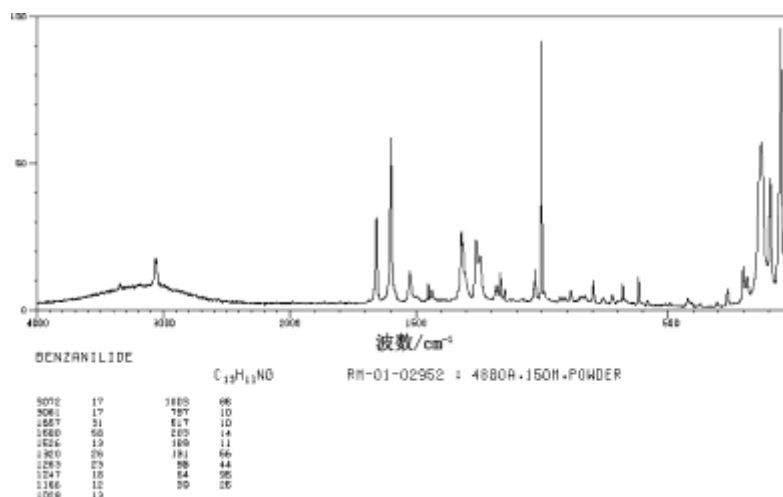
**Interpretation:** Single spot indicates **purity** of the synthesized compound.

## Summary

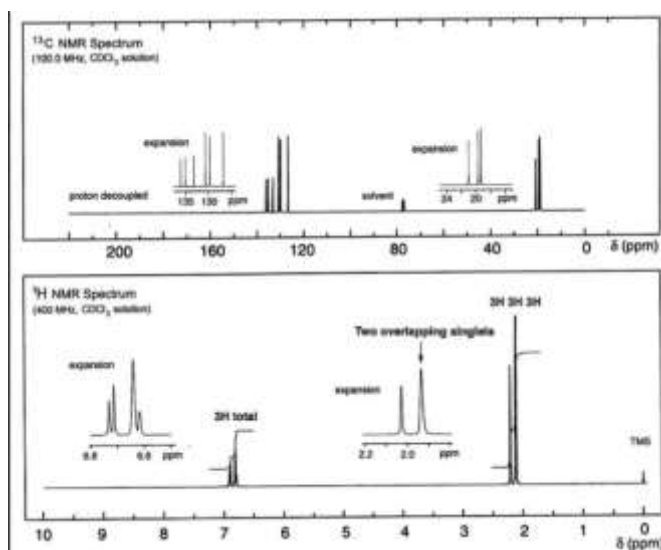
The combination of **melting point determination, IR, NMR, MS, elemental analysis, and TLC** allows unambiguous identification of benzanilide. Key confirming features: **amide carbonyl (C=O), N-H stretch, aromatic protons, and correct molecular weight.**

## Spectral Data :

### 1.<sup>1</sup>H NMR Peaks for Benzanilide (in CDCl<sub>3</sub>):

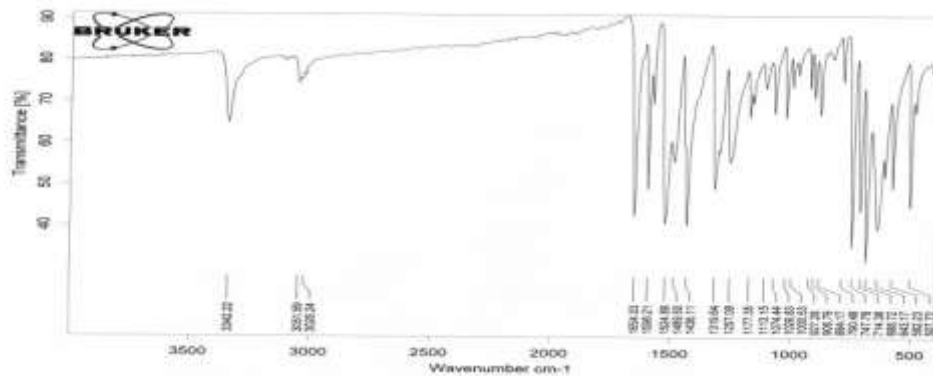


### 3. <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, Schematic)





### 3. IR Spectrum of Benzanilide (Schematic)



### Conclusion:

The **Beckmann rearrangement** provides a reliable and practical method for synthesizing benzanilide from benzene. The overall reaction sequence — Friedel–Crafts acylation, oxime formation, and rearrangement — is straightforward, yields the desired product efficiently, and can be adapted for industrial and laboratory synthesis of aromatic amides.