PW N° 3 : Preparation and Characterization of Acetanilide

1. Objective

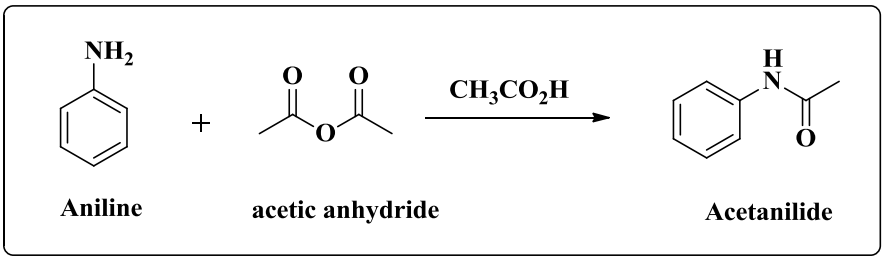
To prepare acetanilide by acetylation of aniline using acetic anhydride, purify the product by recrystallization, and characterize it using melting point and spectroscopic methods (IR, TLC).

2. Introduction

Acetanilide, also known as **acetylaniline** or **acetoaminobenzene**, is an aromatic amide historically recognized for its analgesic and antipyretic properties. It belongs to the same chemical family as paracetamol (acetaminophen) and was once widely used as a pain reliever and fever reducer under the trade name Antifebrin®.

However, in 1948, researchers Julius Axelrod and Bernard Brodie demonstrated that acetanilide exhibits significant toxicity, particularly affecting the liver and kidneys. Subsequent studies also revealed that it can cause methemoglobinemia, a potentially fatal condition characterized by elevated levels of methemoglobin, a non-functional form of hemoglobin in the blood.

Due to these toxic effects, the medical use of acetanilide has been discontinued. Nevertheless, the compound remains of educational and industrial importance, serving as a model compound for demonstrating amide formation reactions (acetylation of aromatic amines) and as a precursor in organic synthesis.



3. Materiel and products

Materiel	products
- Round-bottom flask - pipettes (for liquid volumes) - Heating setup (hot plate or water bath) - Erlenmeyer flask (for crystallization) - Glass rod (to induce crystallization) - Filter setup (Büchner funnel or simple filtration apparatus) - Filter paper - Watch glass (pre-weighed) - Balance (for weighing) - Beaker(s) (for water and ice preparation) - TLC plate and TLC chamber	- Aniline ($C_6H_5NH_2$) - Acetic anhydride $(CH_3CO)_2O$) - Acetic acid (CH_3COOH) - Distilled water (H_2O) - Crushed ice (ice water) - Dichloromethane (CH_2Cl_2) - Acetone

4. Experimental procedure

1. In a flask, introduce **10 mL** of **aniline** and a few grains of pumice stone.
2. Carefully add **15 mL of acetic anhydride** and **20 mL of acetic acid** (serving as solvent and by-product).
3. Heat gently to boiling for 15 minutes.
4. After heating, allow the reaction mixture to cool slightly, then pour it into an Erlenmeyer flask containing 150 mL of distilled water and crushed ice.
5. A white solid appears (crystallization can be initiated by scratching the bottom of the flask with a glass rod).
6. Filter the mixture. Press the crystals to start drying them.

7. Place the crystals in a pre-weighed watch glass, then dry them completely in an oven.

Thin Layer Chromatography (TLC):

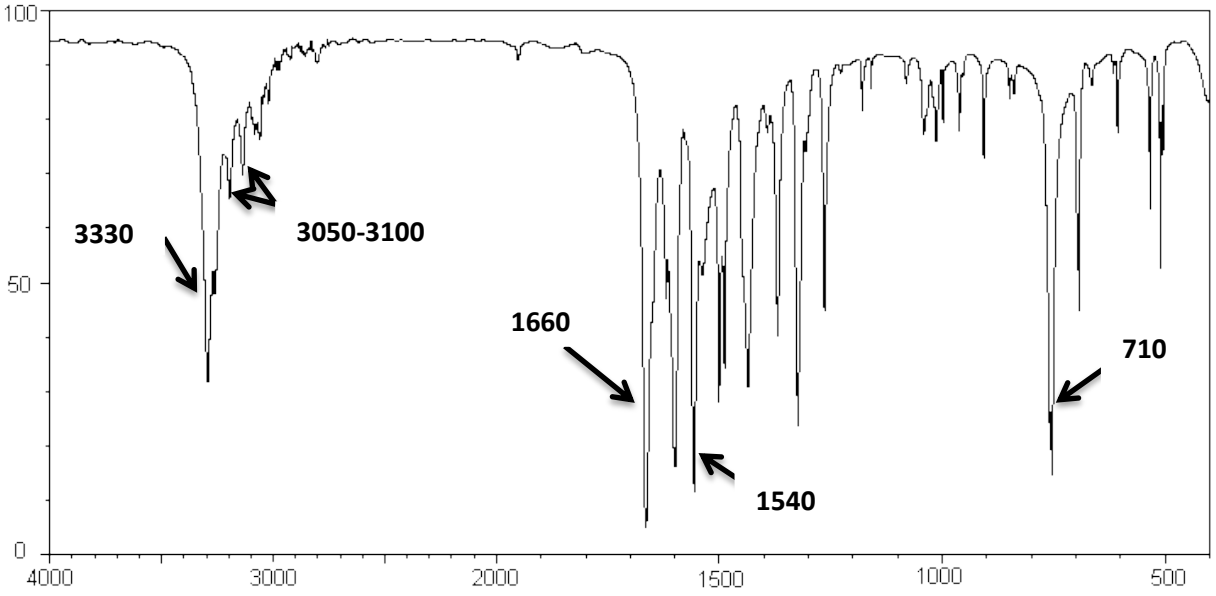
Eluent: dichloromethane/acetone (50/50 by volume).

Recrystallization:

Acetanilide is soluble in hot water and insoluble in cold water.

5. Questions

1. What role does acetic acid play in this reaction?
2. What is the purpose of heating gently for 15 minutes during the reaction?
3. Why is the reaction mixture poured into ice-cold water after heating?
4. Why must the crystals be filtered and dried before characterization?
5. Why is recrystallization performed using hot water?
6. How can TLC (Thin Layer Chromatography) confirm the purity of acetanilide?
7. What is the expected Rf value trend between aniline and acetanilide? (Which will have the higher Rf and why?)
8. Which functional groups are expected to appear in the IR spectrum of acetanilide?
9. Calculate the theoretical yield of acetanilide if you start from 10 mL of aniline.
10. If your experimental yield is lower than expected, list possible sources of loss or error.
11. Explain how this experiment demonstrates the concept of functional group transformation in organic chemistry.
12. Explain how **crystallization** occurs in this experiment.
13. Analyze the IR spectrum and identify the main absorption bands corresponding to the functional groups in acetanilide.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