Lab N °02: Synthesis / Purification by recrystallization of benzoic acid

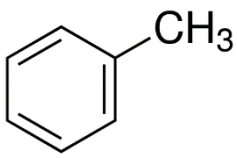
Objectives

- We have a mixture of benzoic acid and toluene in ether. Using the chemical and physical properties of the various components, we aim to separate and purify the benzoic acid.
- Acquire basic techniques in organic chemistry.

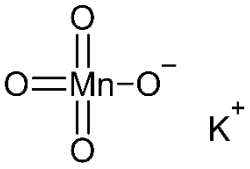
Introduction

Benzoic acid, with the chemical formula $C_7\ H_6O_2$, is a white crystalline solid characterized by its aromatic odor. Widely employed as a food preservative, it effectively inhibits the growth of bacteria, molds, and yeasts in acidic food products and beverages. It exhibits limited solubility in water but readily dissolves in organic solvents such as ether, ethanol, and acetone.

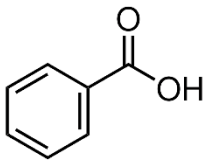
Benzoic acid can be synthesized from toluene using potassium permanganate $4KMnO_4$) through a process known as oxidation.



Toluene
$C_6H_5CH_3$



Potassium Permanganate
$KMnO_4$



Benzoic Acid
$C_6H_5CH_3COOH$

Materials & Equipment:

Materials	Equipment	
potassium permanganate KMnO4.	Electronic Balance	Büchner
Distilled water	Refrigerant	Filter papers
Toluene	Double necked flask	Separatory funnel
Methanol	Hot plate magnetic stirrer	PH paper
Conc hydrochloric acid (HCl)	Magnetic stirring bar	Fume Hood
	100 mL Erlenmeyer	Watch glass

Procedure

1. weigh 10 g of potassium permanganate $KMnO_4$.
2. In a 250ml double-necked flask, put 10g of permanganate and 50mL of distilled water. Then, bring the mixture to a gentle boil
3. Gradually, introduce 8mL of toluene while maintaining the boiling for 2 hours.
4. Add 5 mL of methanol and continue heating for 5 minutes.
5. Stop heating while maintaining water circulation and allow to cool.
6. Remove the flask from the reflux setup and cool it with water.
7. Filter the contents of the flask.
8. Pour the filtrate into a beaker placed in ice. Gently add a concentrated aqueous solution of hydrochloric acid (HCl).
9. A white precipitate of benzoic acid forms.
10. Once the mixture is well cooled, perform a Büchner filtration.
11. Thoroughly dry the obtained solid and transfer it to a watch glass.
12. Weigh the solid once dry.
13. Recrystallize the benzoic acid in a water/methanol mixture.

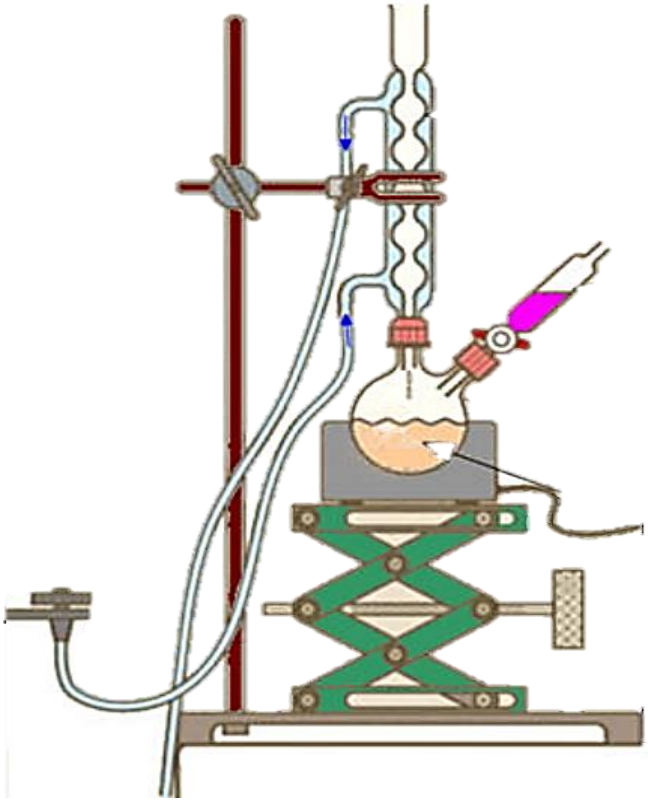


Figure 1 Setup and protocol

Questions

1. What is the topological representation of toluene?
2. What is the role of KMnO4?
3. What is the interest of hydrochloric acid?
4. How can the synthesized product be characterized?
5. Write the equations for oxidation and reduction reactions.
6. Write a balanced redox equation for the reaction.