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Cannizzaro Reaction

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Part II

Name of Experiment : **CANNIZZARO REACTION.**

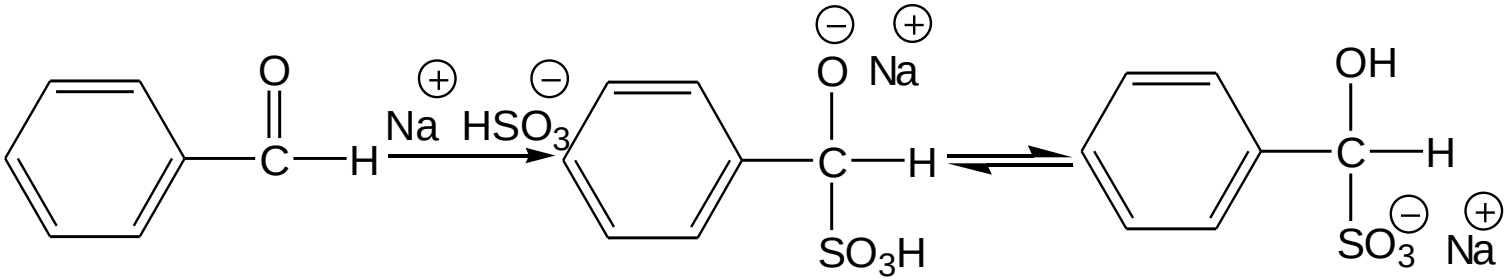
Aim of Experiment : Isolation of **BENZOIC ACID** & **BENZYL ALCOHOL**

Procedure :

1- ***Add 30 mL of H_2O to completely dissolve the Potassium benzoate.***
2- ***Pour the liquid into a separatory funnel.***
3- ***Rinse the bottle with 10 mL of ether & add this ether to the solution in the separatory funnel.***
4- ***Shake well & separate the lower aq. layer from upper ethereal layer.***
5- ***Extract the aq. layer with 10 mL ether. (two times)***

A- Isolation of Benzyl alcohol :

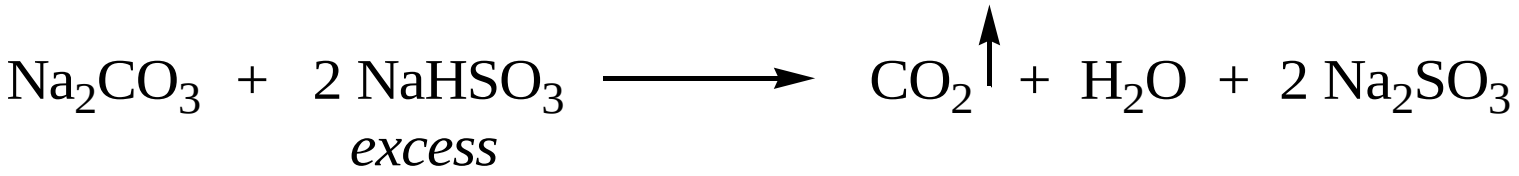
1- **Combine the ethereal extracts.**
2- **Distil the ether on a water bath until 10 – 15 mL of liquid remains.**
3- **Cool the remaining liquid and transfer it to a separatory funnel.**
4- **Shake the ether layer with 3 mL of saturated Sodium bisulfite solution, $NaHSO_3$ *, & separate the oily liquid from the aq. $NaHSO_3$ solution, (two times).**



a-hydroxy benzyl sodium sulfonate

* *To remove any unreacted Benzaldehyde which may be present.*

5- Wash the oily liquid with 5 mL of 10 % Na_2CO_3 **



** *To ensure complete removal of unreacted $NaHSO_3$*

6- Wash with 5 mL H_2O .

7- Dry the oily liquid over anhydrous Magnesium sulfate, Mg_2SO_4 .

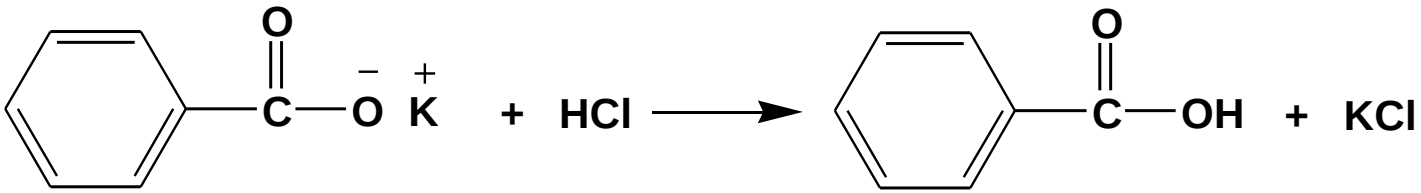
8- Filter the solution into a small distilling flask & distil off the ether on a water bath.

9- Attach a short air – cooled condenser & distil the Benzyl alcohol .

10- Collect the material boiling at 204 – 207 ^oC .

B- Isolation of Benzoic acid :

1- Pour the alkaline aqueous layer with stirring into a mixture of 20 mL conc. HCl + 20 mL H_2O + 20g of crushed ice .



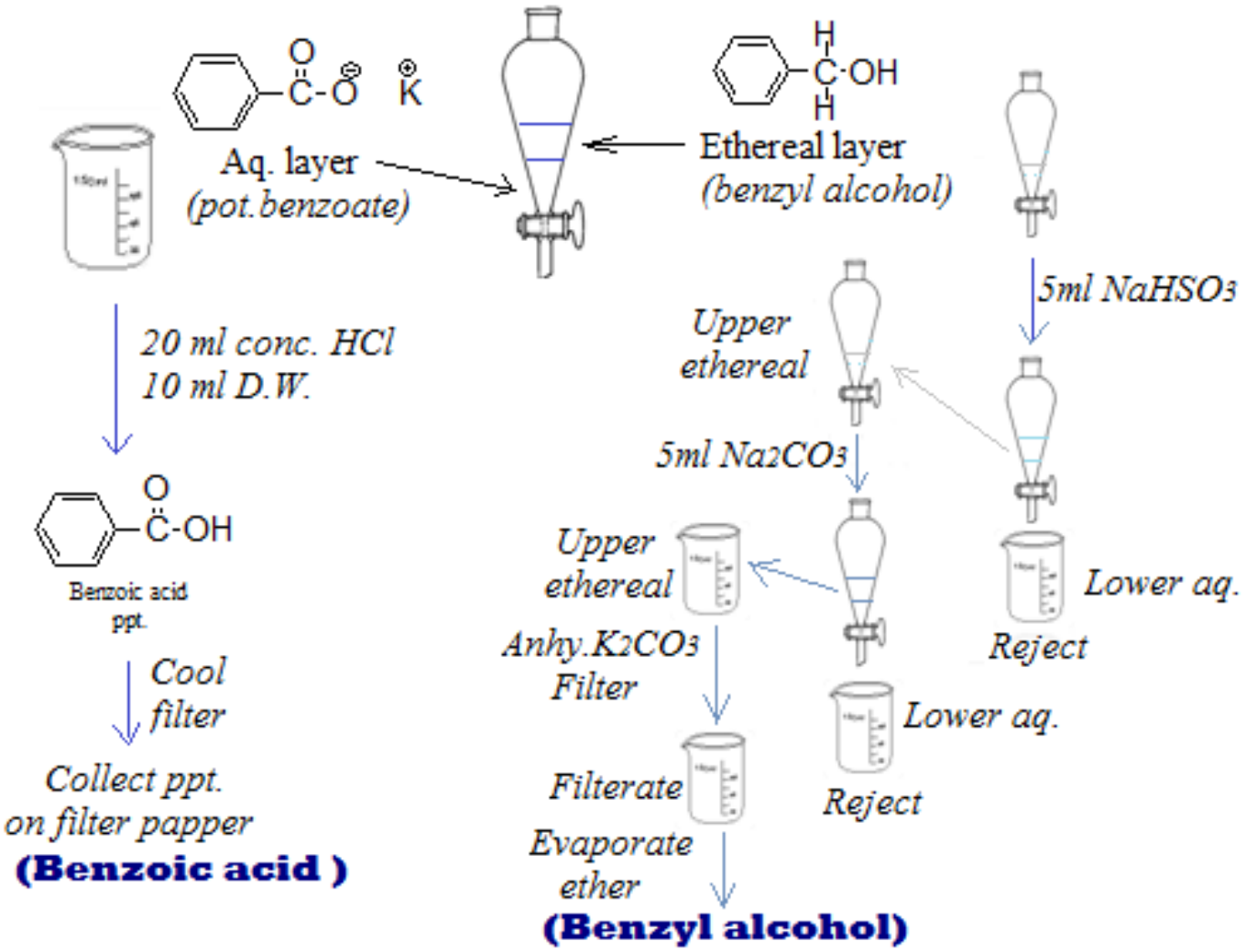
Potassium benzoate
(*Soluble in water*)

Benzoic acid
(*Insoluble in cold water*)

2- Filter the ppt. (Benzoic acid) at the pump.

3- Wash the ppt. with little cold H_2O .

4- Drain & recrystallize from boiling H_2O .
(colorless crystals of Benzoic acid, m.p. 121 ^oC) .


Aq. layer
(pot.benzoate)
Ethereal layer
(benzyl alcohol)
20 ml conc. HCl
10 ml D.W.
Benzoic acid
ppt.
Cool
filter
Collect ppt.
on filter papper
(Benzoic acid)
5ml NaHSO3
Upper
ethereal
5ml Na2CO3
Lower aq.
Reject
Upper
ethereal
Anhy.K2CO3
Filter
Lower aq.
Filterate
Reject
Evaporate
ether
(Benzyl alcohol)

Post Lab. Exercises :

1- In extraction by ether & water , which one of them will be the upper layer ? And why ?

2- How can we get rid of the excess unreacted Benzaldehyde ?

3- Explain why , 10% Na_2CO_3 solution is used for washing the ether layer ?

4- How can we purify the synthesized Benzoic acid ? Explain .

References

* Robert T. Morrison , Robert N. Boyd: *" Aldehydes and Ketones"* . **Organic Chemistry**, (6th) edition , Prentice - Hall Inc.

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* Samira Finjan Hassan, Amer Nadem, May Mohammed Jawad , **A Laboratory manual on Practical Medical Chemistry for 4th year students**, *University of Baghdad , College of Pharmacy , Department of Pharmaceutical Chemistry, 2010.*

* Vogel, Arthur,**Textbook of Organic Chemistry**,4th edition.

* John E. McMurry,**Organic Chemistry**, 8th edition, 2012