2015 - 2016

RECRYSTALLIZATION OF SALICYLIC ACID

Assistant Lecturer

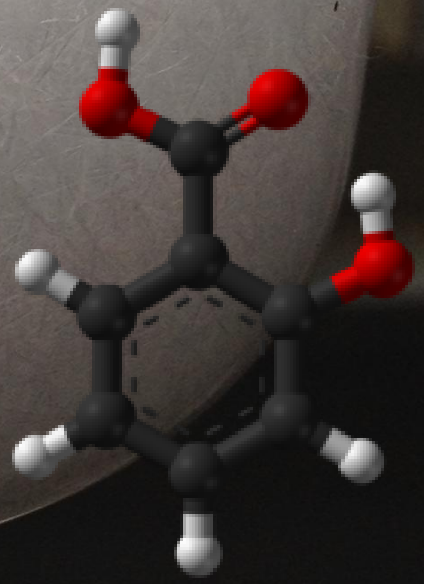
Sahar Mohammed Shakir

Assistant Lecturer

Abdul Hafeedh Hameed

Assistant Lecturer

Ali Basim



Solid organic cpd.s when isolated from organic reaction are impure; they are contaminated with small amounts of other cpd.s produced along with the desired product.

The purification of impure crystalline compound is usually done by **Recrystallization** from a suitable solvent or a mixture of solvents.

Purification of solids by recrystallization is **based upon** differences in their solubility in a given solvent or a mix. of solvents.

Desirable solv. characteristics for Recrystallization:

1- Chemically inert toward the solute.

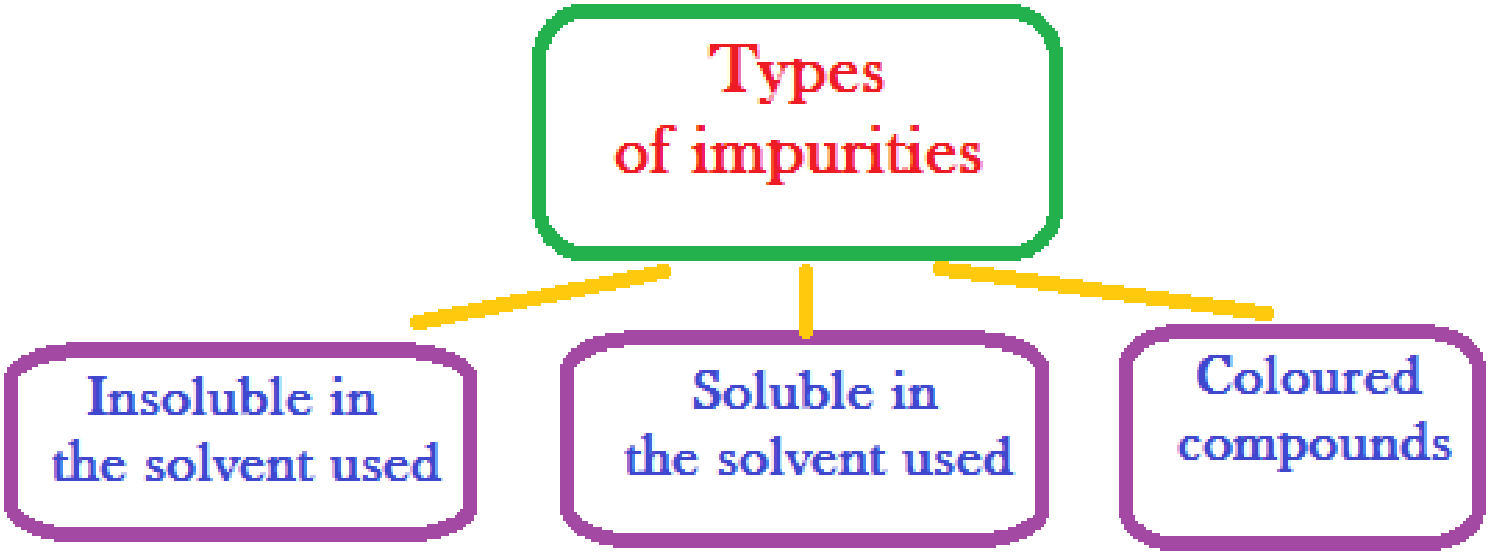
2- It should dissolve the solute to be purified readily at or near it's boiling point, but sparingly at the lab. temp. or below (0 – 25 ^{o}C).

3- It should dissolve the impurities readily or not at all.

4- It should be capable of easy removal from the crystals of the purified cpd. , (i. e.) possess a relatively low b.p.

5- It should yield well-formed crystals of the cpd.

6- If 2 or more solvents appear to be equally suitable for recrystallization the final selection will depend up on such factors as ease of manipulation, lower toxicity, Less flammability, & Lower cost.



Simple Recrystallization process consist of:

1- Dissolving the impure substance in some suitable solvent at or near the boiling point.

2- Filtering the hot solution from the particles of insoluble material & dust, (Hot filtration).

3- Allowing the hot solution to cool thus causing the dissolved substance to crystallize out.

4- Separating the crystals from the supernatant soln.

How could we choose a good solvent :

Practically,

Take **0.1g** of a pure sample of cpd. to be purified & try to dissolve it in **1ml** of solvent,

if it;

Dissolves in the **Cold** solvent → The solvent is **Not suitable**

If it;

Dissolves in the **Hot** solvent → The solvent is **Suitable**

& If it;

Doesn't dissolve in **Hot & Cold** solvent → The solvent is **Not suitable**

just an Example

Results of solubility tests for cpd. (A) are shown in table (*g/ml*).

Solvent	Water	Ethanol ✓	Diethyl ether
Cold	20	3	5
Hot	30	25	5

Which solvent will you choose to recrystallize cpd. A?

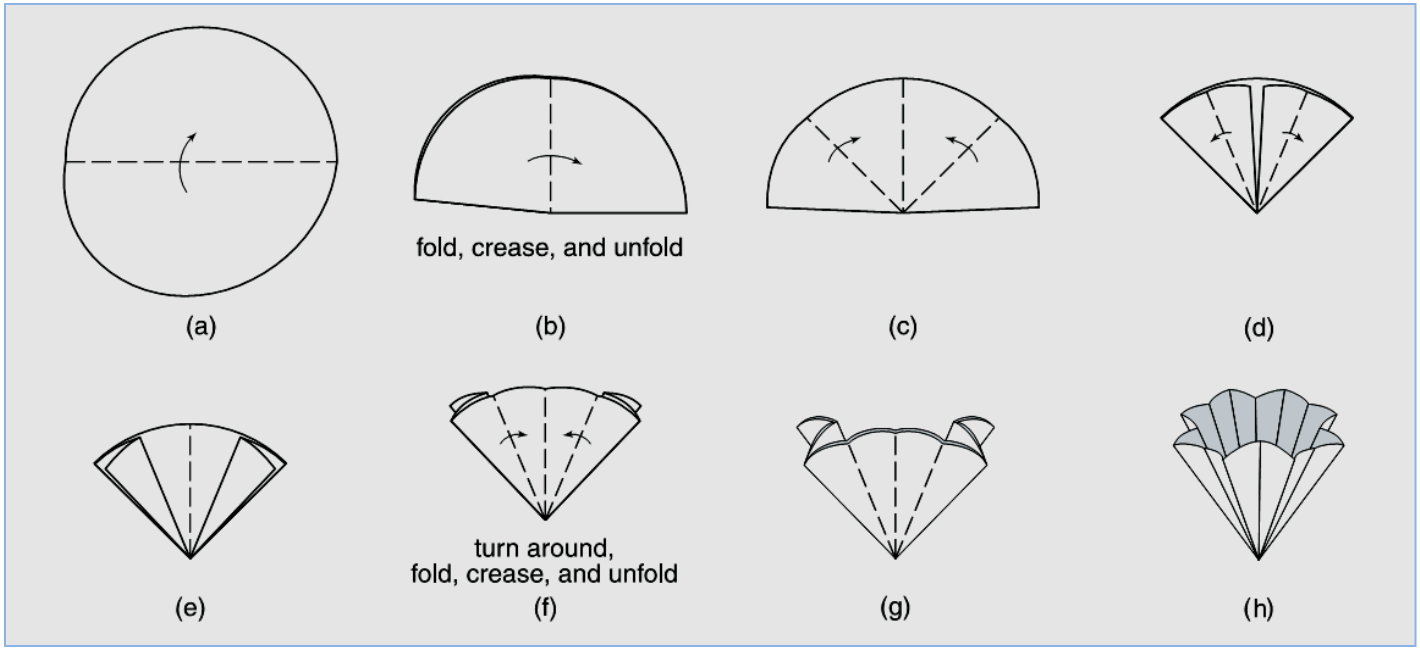
Common solvents used For recrystallization:

Solvent	b.p. (°C)	
Water (distilled)	100	To be used whenever suitable
Methanol*	64.5	Flammable; toxic
Ethanol	78	Flammable
Industrial spirit	77–82	Flammable
Rectified spirit	78	Flammable
Acetone	56	Flammable
Ethyl acetate	78	Flammable
Acetic acid (glacial)	118	Not very flammable, pungent vapours
Dichloromethane (methylene chloride)*	41	Non-flammable; toxic
Chloroform*	61	Non-flammable; vapour toxic
Diethyl ether	35	Flammable, avoid whenever possible
Benzene*†	80	Flammable, vapour highly toxic
Dioxane*	101	Flammable, vapour toxic
Carbon tetrachloride*	77	Non-flammable, vapour toxic
Light petroleum	40–60	Flammable‡
Cyclohexane	81	Flammable

Filtration of hot solution:

The boiling and hot solution must be rapidly filtered before undue cooling using *fluted filter paper* to:

- **Increase the surface area of filtration for fast filtration.**
- **The fluting creates gaps between the filter paper and the funnel in which it is resting,**
  thus greatly increasing the speed of solvent flow through the paper.



Using Charcoal:

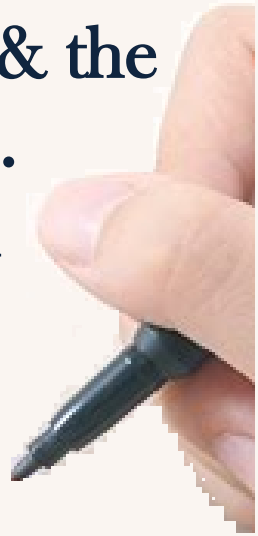
Samples to be purified may contain soluble colored impurities that may cause the soln. & the crystals to be colored.

Up on Recrystallization these impurities dissolves in the boiling solvent & adsorbs on the crystals produced up on cooling yielding a colored product.

Activated charcoal

composed of fine carbon particles with a large active surface area on which the colored impurities will be adsorbed.

Charcoal is added to the hot soln. before boiling & the soln. is kept hot at or near the b.p. for about 3 – 5 min. with shaking to wet the charcoal, the solution is then filtered through a fluted filter paper.



Notes about using activated charcoal:

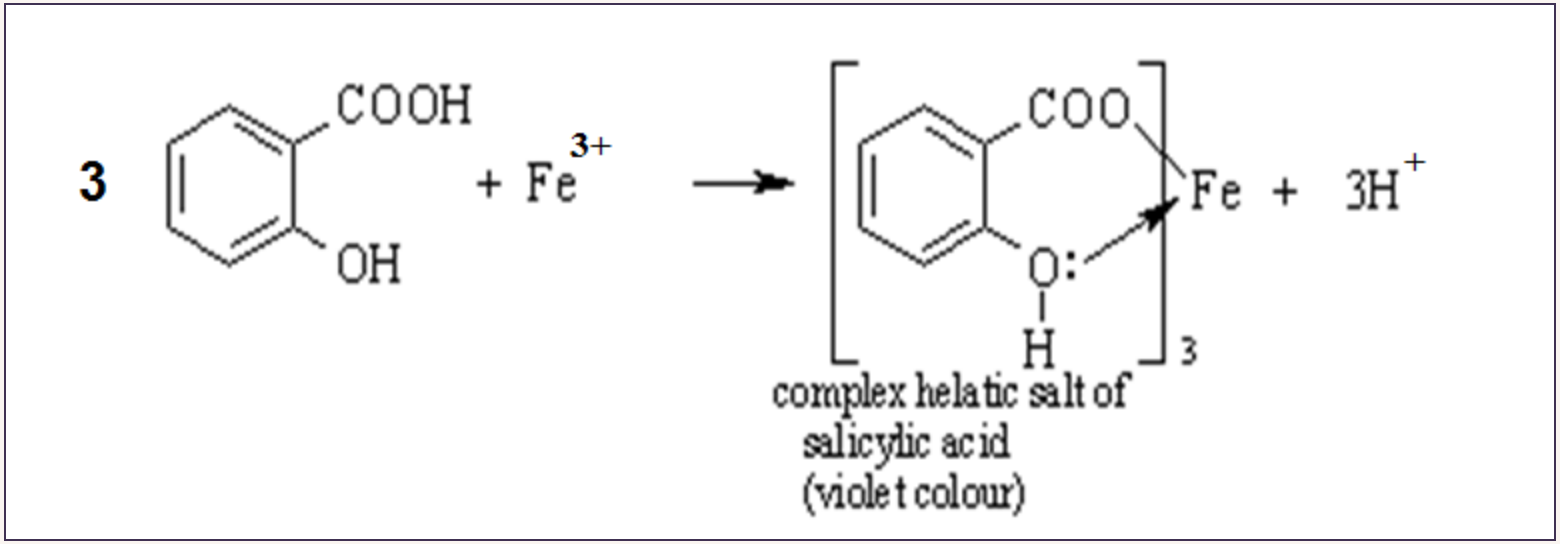
- An excessive quantity of decolorizing charcoal should be avoided since it may adsorb some of the cpd. which is being purified.

- Charcoal should not be added to a superheated solution or at the b.p. of the solvent because it's particles function as thousands of boiling chips causing the solution to boil over and foam.

Charcoal is **Not** used for recrystallization of **phenolic cpd.s**

Because,

They contain ferric ions (Fe^{3+}) that upon heating the solution for some times it can react with the phenolic −OH group forming red – violet colored complexes thus impairing the purification process.



Recrystallization using mixed solvents:

It is applied when our cpd. is readily soluble in a solvent at room temp. & insoluble in other solvent, The 2 solvents must be miscible with each other as

Alcohol & water, ether & pentane

glacial acetic acid & water

Procedure :

1- The cpd. is dissolved in the solvent that is soluble in it.
2- Charcoal is used if required.
3- The solution is filtered to get rid of the insol. impurities.
4- The other solvent (in which the cpd. is insoluble), is added to the filtrate gradually until turbidity appears.
5- The mixture is then left a side to facilitate crystallization.

General Notes:

- ***If recrystallization fails to occur , then you should:***

1- Scratch the sides or the bottom of the container below the surface of the solution with a glass rod.

2- Add small crystals of the pure cpd.

3- *Or*, you can evaporate some of the solvent to induce crystallization process.

- **During Hot Filtration,**

  Funnel, Filter paper and the container of the solution should be kept hot throughout the filtration process to prevent the deposition of the crystals on the filter paper or on the neck of funnel therefore it's recommended to wash the filter paper after completing the filtration process with a small amount of hot solvent.

- **Minimum volume of solvent is used to prevent the loss of compound, large volumes of solvent will keep most of the compound dissolved in it.**
- **Drying of the purified substance can be achieved by:**

  air dryer, oven, freeze drying or by using desiccators containing a drying agent such as, *anhydrous calcium chloride* ($CaCl_2$) or *silica gel* (a granular, vitreous & porous form of Silicon dioxide, SiO_2).

Oven

Desiccator

Freeze Dryer Machine

Lyophilizer

Name of Experiment: **Recrystallization of Salicylic Acid**

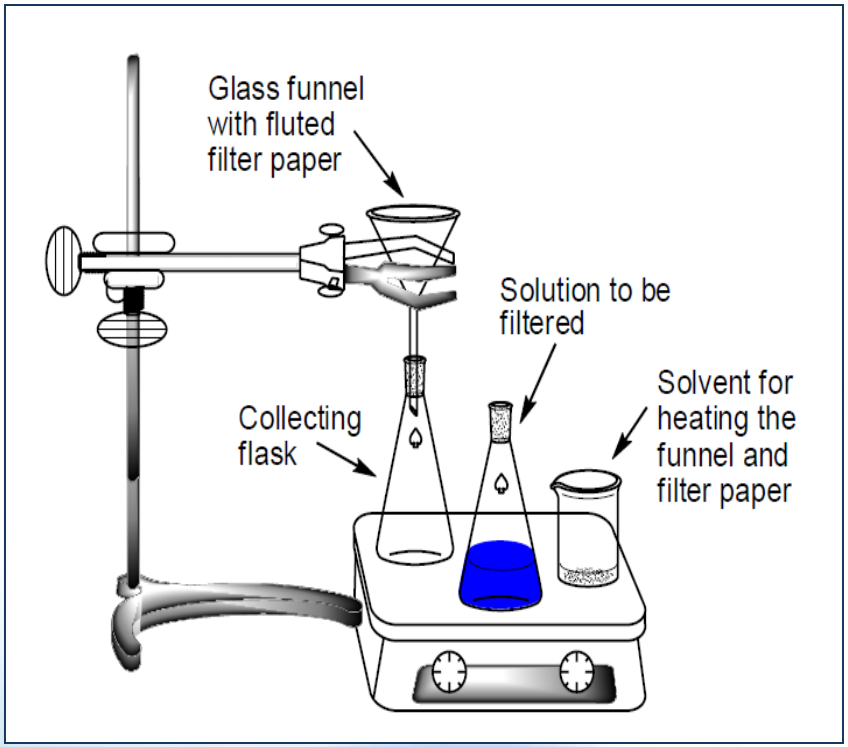
Aim of experiment: **Purification of the synthesized Salicylic Acid.**

Procedure:

1- **Put 1 g of impure Salicylic acid sample in a beaker.**

2- **Try to dissolve it in a minimum amount of hot water with heating.**

3- **Filter the solution while it is hot,**
all the equipments should be kept hot during the first filtration ,
(*Hot filtration*).



4- Cool the filtrate, then Salicylic acid will crystallize.
5- Filter again, (*Cold filtration*).
6- Collect the crystals of S.A. on the filter paper and dry them on oven.

The resulting solid after drying is tested for purity usually by melting point determination, spectroscopic method or by TLC.

If it's found impure it should be recrystallized again from fresh solvent.