Recrystallization of Salicylic acid

Recrystallization

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.

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Choosing a solvent for Recrystallization:

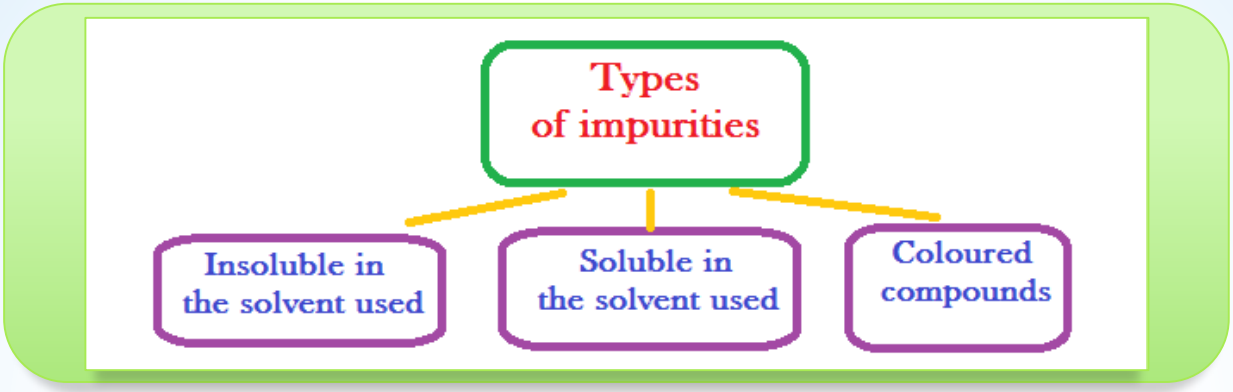
The ideal solvent should :

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- not be flammable , low cost , and of low toxicity .



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).

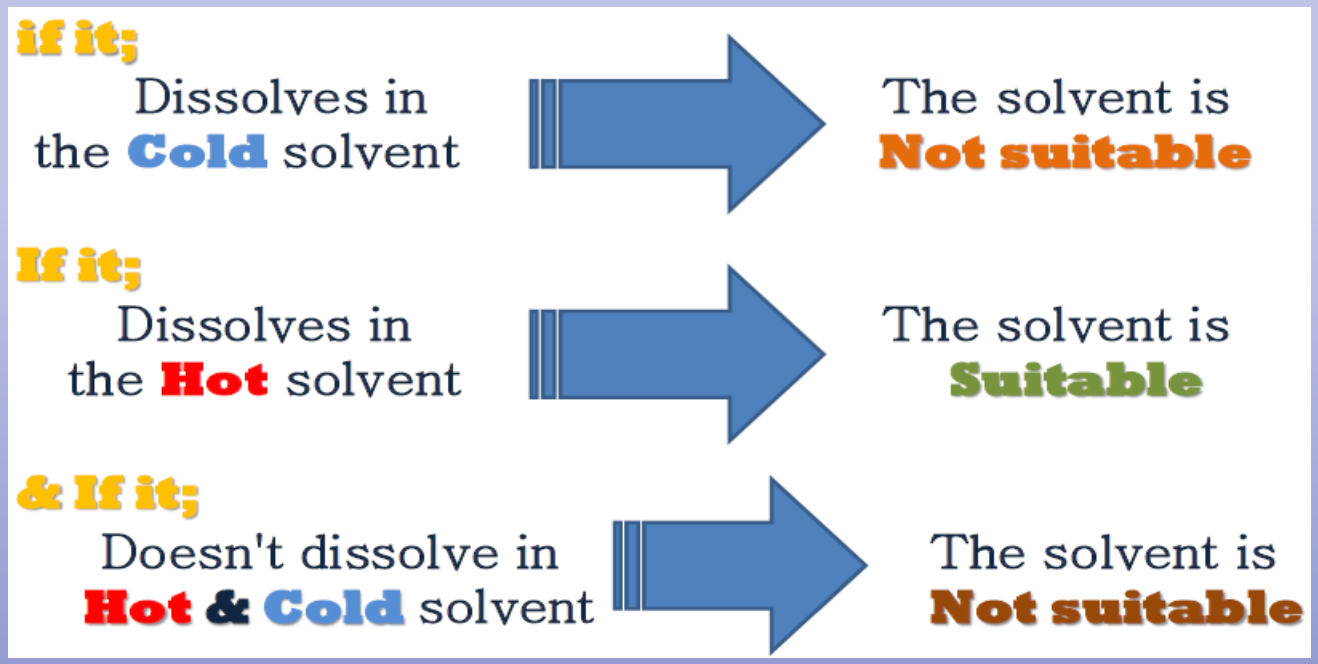
3- Allowing the hot solution to cool thus causing the dissolved substance to crystallize out.

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,



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?

Using Charcoal:

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

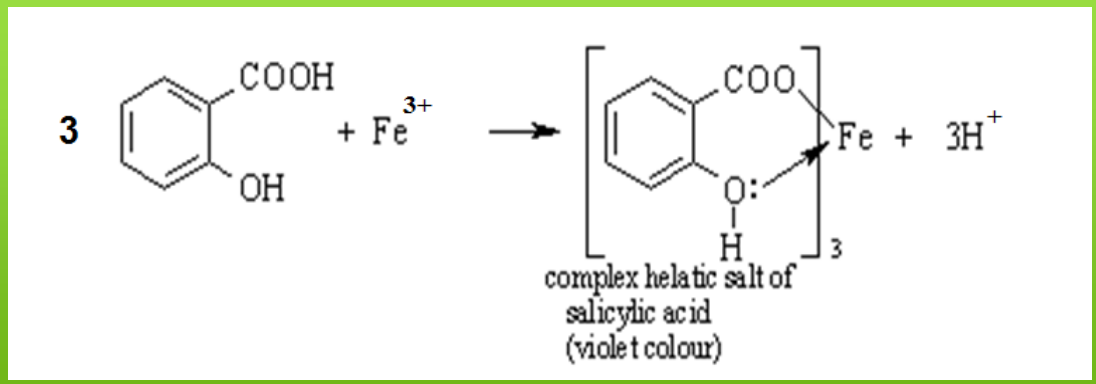
Up on Recrystallization these impurities dissolves in the boiling solvent & adsorbs on the crystals produced up on cooling yielding a colored product.

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.

Q / Charcoal is Not used for recrystallization of phenolic cpd.s

Ans :Because, They contain ferric ions (Fe) 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 .

Name of Experiment: Purification of the synthesized
Aim of experiment: Purification of the synthesized Salicylic Acid.

Procedure :

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

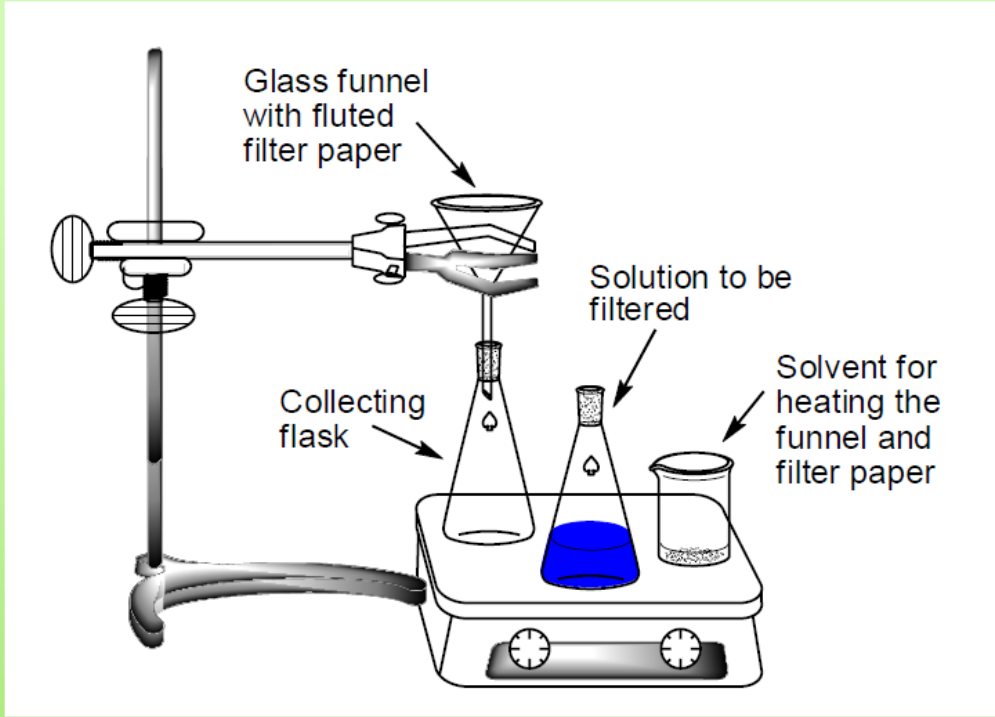
2- Try to dissolve it in a minimum amount of hot water with heating. Continuously

3- Filter the solution while it is hot .

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.


Glass funnel
with fluted
filter paper
Solution to be
filtered
Solvent for
heating the
funnel and
filter paper
Collecting
flask

Calculations :

$$\text{Percent pure substance \%} = \frac{\text{pure substance wt.}}{\text{Sample wt .}} \times 100$$

$$\text{Percent impure substance \%} = 100 - \text{Percent pure substance}$$