



Recrystallization of Salicylic acid



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Table of contents

01

Introduction

02

**Recrystallization
process**

03

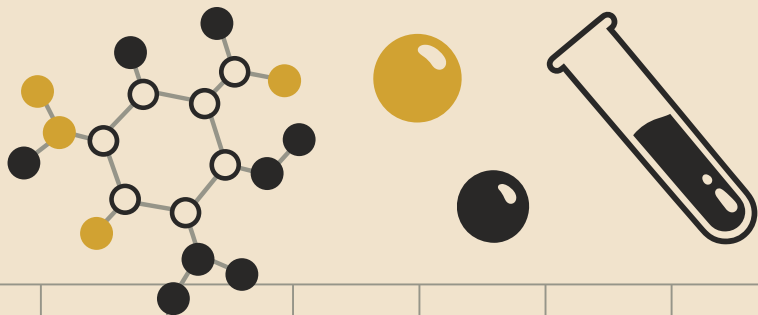
Using charcoal

04

**Recrystallization
using mixed
solvents**

05

**General notes &
experiment**





Introduction

- ❑ **Solid organic** compounds when isolated from organic reaction are impure; they are contaminated with small amounts of other compounds 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 **mixture of solvents**.



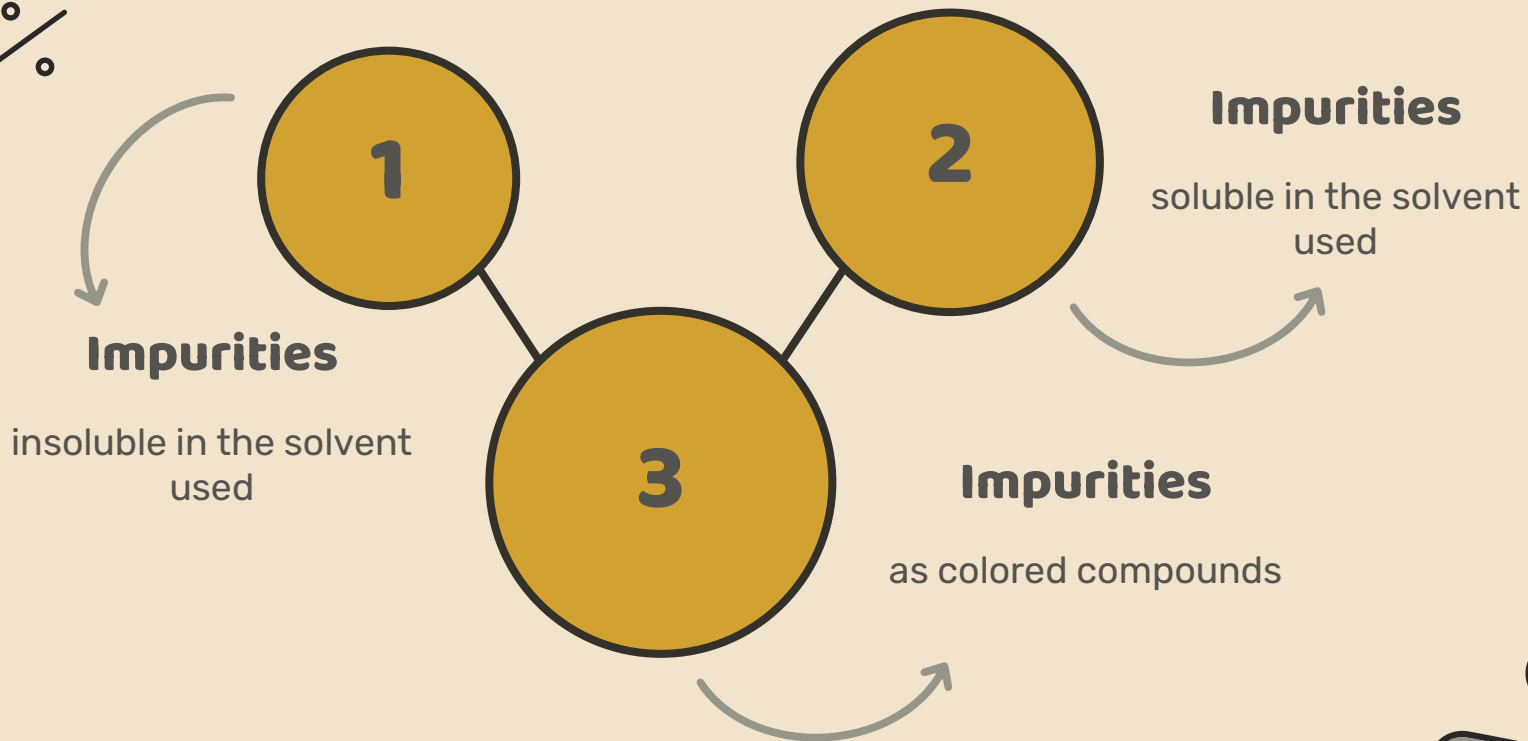
Desirable solvent characteristics for Recrystallization



1. **Chemically inert** toward the solute.
2. It should **dissolve the solute** to be purified readily at or near its boiling point, but sparingly at the lab. temp. or below (0 – 25 oC).
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 compound , (i.e. possess a relatively low boiling point)
5. It should yield **well-formed crystals** of the compound.
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.**



Types of impurities





Simple Recrystallization process



consist of:

01
Dissolving the impure substance in some suitable solvent at or near the boiling point.

01

02

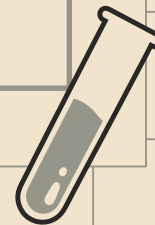
02
Filtering the hot solution from the particles of insoluble material & dust, (Hot filtration)

03
Allowing the hot solution to cool thus causing the dissolved substance to crystallize out.

03

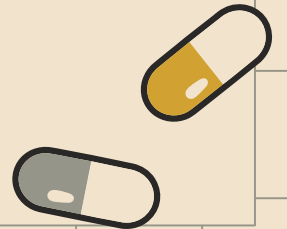
04

04
Separating the crystals from the supernatant solution.





**How could we
choose a good
solvent?**





Choosing a good solvent

Practically

Take 0.1 g of a pure sample of compound to be purified & try to dissolve it in 1 ml of solvent;

01

If the sample dissolves in the cold solvent

The solvent is not suitable

02

If the sample dissolves in the hot solvent, but not in the cold one

The solvent is suitable

03

If the sample Doesn't dissolve in the Hot & Cold solvent

The solvent is not suitable

Example

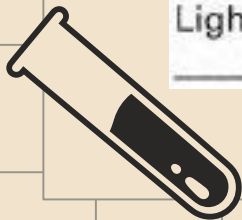
Results of solubility tests for compound (X) are shown in table (g/ml) below, which solvent will you choose to recrystallize the compound?

| Solvent | Hexane | Water | Ethanol |
|---------|--------|-------|---------|
| Cold | 35 | 5 | 4 |
| Hot | 45 | 6 | 30 |



Common solvents used For recrystallization

| Solvent | b.pt. (°C) | Polarity | Miscibility with water | Comments: class of organic compounds recrystallized |
|-----------------|---------------|----------|---------------------------|--|
| Water | 100 | V. high | Yes | Always use when suitable: salts, aromatic acids |
| Ethanol | 78 | High | Yes | Flammable: alcohols, acids, amides |
| Propanone | 56 | High | Yes | Flammable: carbonyl compounds |
| Ethyl ethanoate | 78 | Medium | No | Flammable: esters |
| Dichloromethane | 41 | Medium | No | Toxic: halogen compounds, ethers |
| Toluene | 110 | Low | No | Flammable: hydrocarbons |
| Light petroleum | 40-60 | V. low | No | Flammable: hydrocarbons |

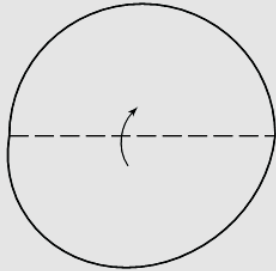
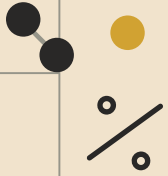


Filtration of hot solution

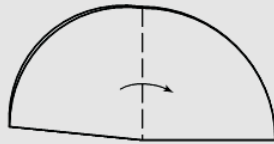


The boiling and hot solution must be rapidly filtered before undue cooling using fluted filter paper to:

- 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.

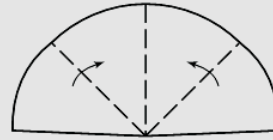


(a)

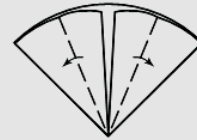


fold, crease, and unfold

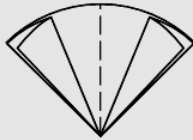
(b)



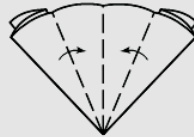
(c)



(d)

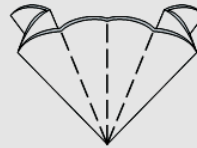


(e)

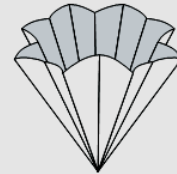


turn around,
fold, crease, and unfold

(f)



(g)



(h)



**Have a
break**



Using Charcoal



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



Upon 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.



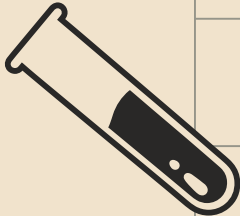

Charcoal is added to the **hot solution** before boiling & the solution is kept hot at or near the b.p. for about 3 – 5 min. with **shaking** to wet the charcoal, the solution is then filtered through a **fluted filter paper**.



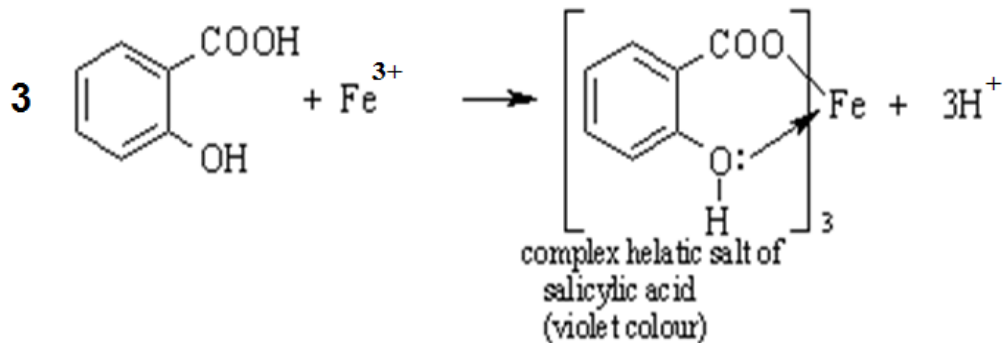
—Notes about using activated charcoal:

- ❑ An excessive quantity of decolorizing charcoal should be avoided since it may adsorb some of the compound which is being purified.
- ❑ Charcoal should not be added to a superheated solution or at the b.p. of the solvent because its particles function as thousands of boiling chips causing the solution to boil over and foam.
- ❑ Charcoal is Not used for recrystallization of phenolic compounds. **Why?!!**





Charcoal is Not used for recrystallization of phenolic compounds because, they contain ferric ions (Fe^{3+}) that upon heating the solution for some times it can react with the phenolic $-\text{OH}$ group forming red - violet colored complexes thus impairing the purification process.





Recrystallization using mixed solvents

- ❑ It is applied when our compound 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



General procedure of recrystallization

1



The compound is dissolved in the solvent that is soluble in it. (Charcoal is used if required).

2



The solution is filtered to get rid of the insol. impurities.

3

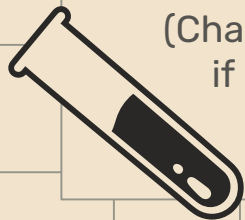


The other solvent (in which the cpd. is insoluble), is added to the filtrate gradually until turbidity appears.

4



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



General notes

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.



General notes

- 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).

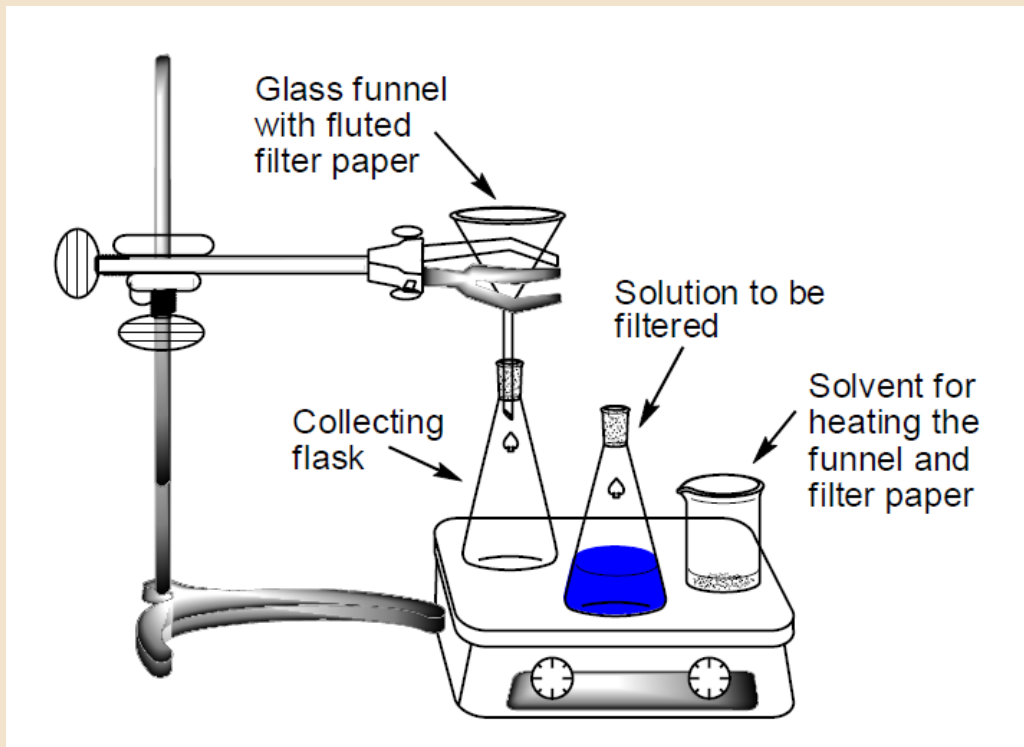


Desiccator




Name of Experiment: Recrystallization of Salicylic Acid

Aim of experiment: Purification of the synthesized Salicylic Acid



Procedure



Put 1 g of impure Salicylic acid sample in a beaker

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

Filter again, (Cold filtration).

01

02

03

04


05

06

Try to dissolve it in a minimum amount of hot water with heating

Cool the filtrate, then Salicylic acid will crystallize

Collect the crystals of S.A. on the filter paper and dry them on oven.





Notice that:

- 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.

Thanks!

For your listening !

