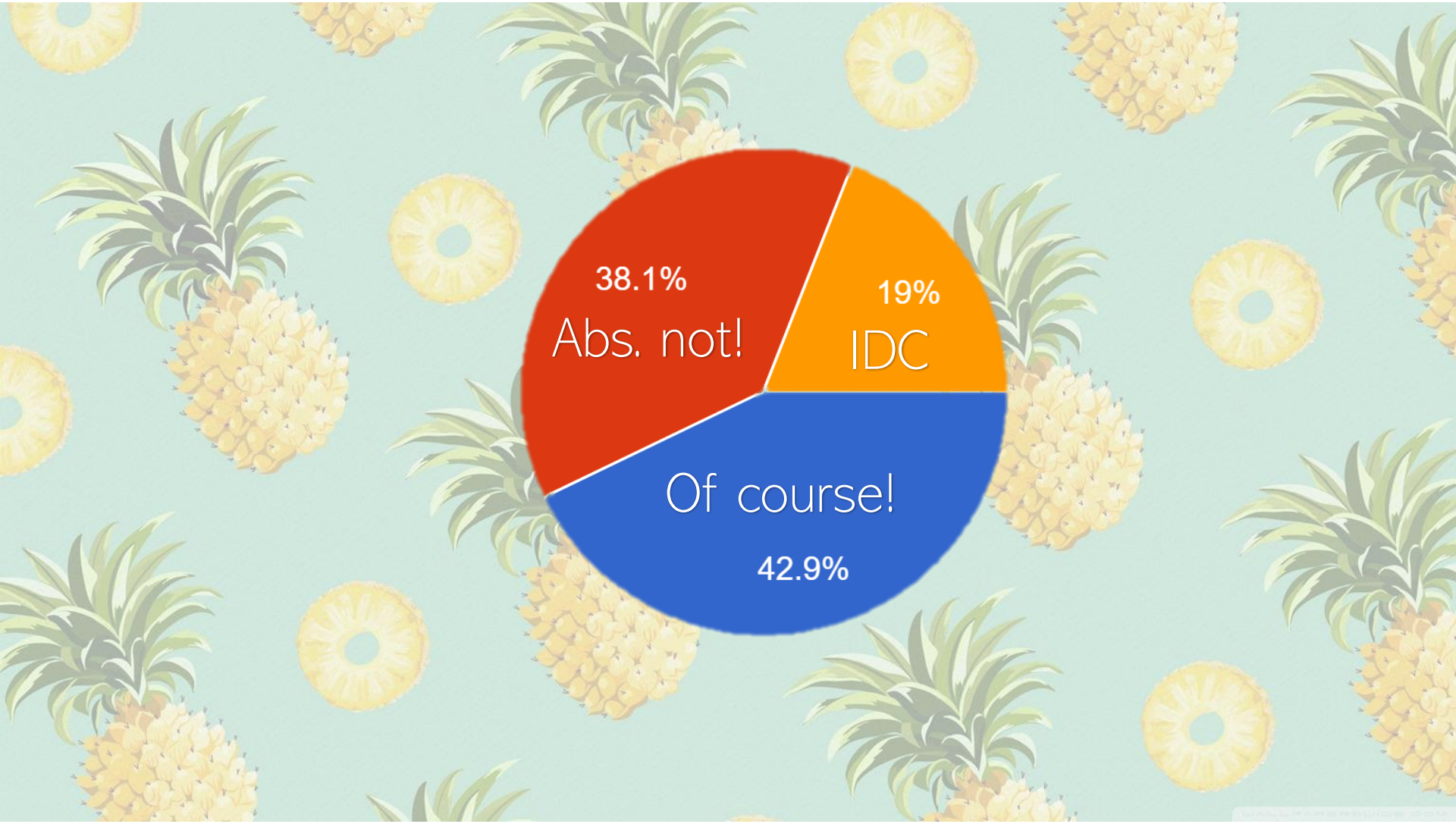
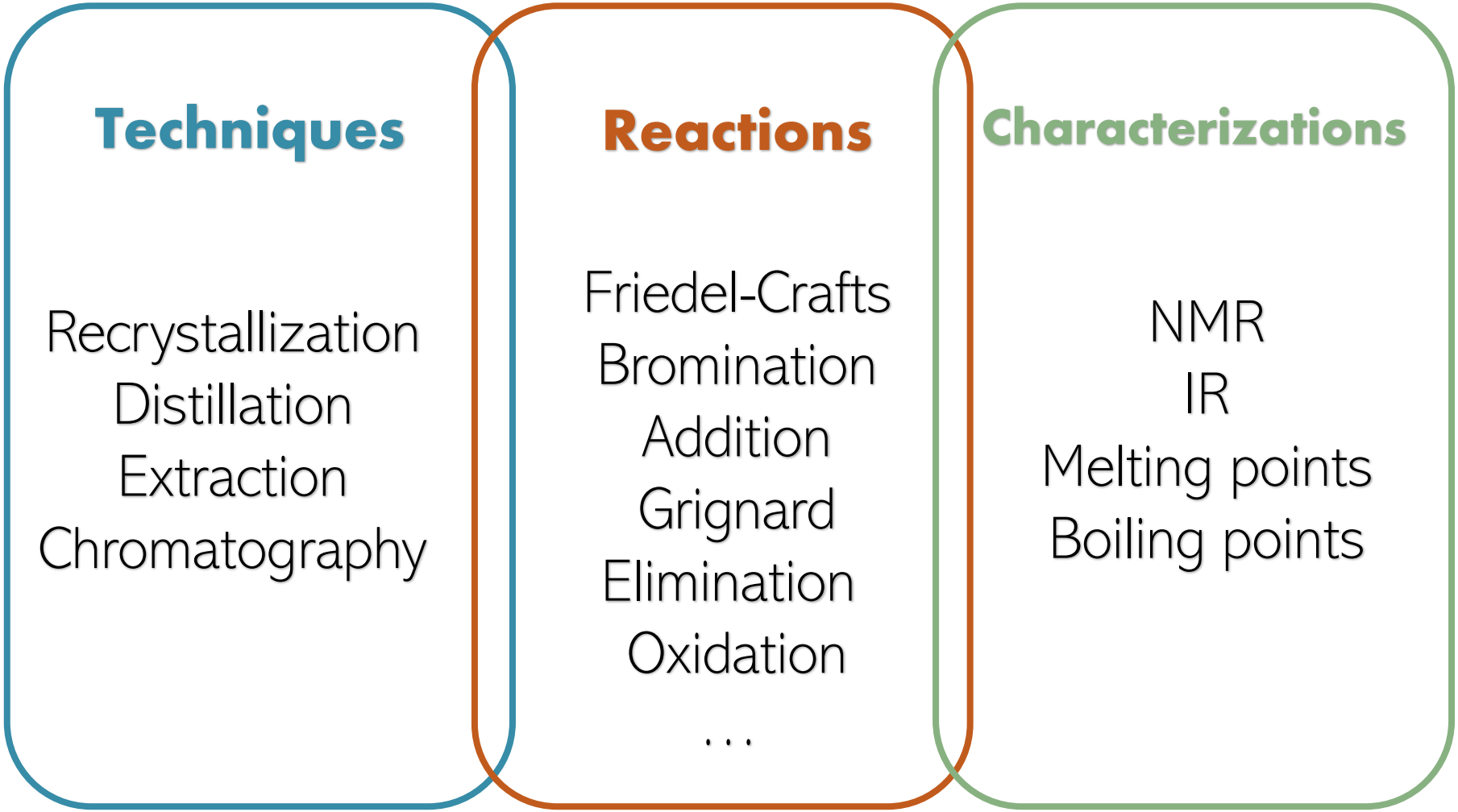
Pineapple on pizza?




Techniques
Recrystallization
Distillation
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Chromatography
Reactions
Friedel-Crafts
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Addition
Grignard
Elimination
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. . .
Characterizations
NMR
IR
Melting points
Boiling points

Recrystallizations & Extraction
Chem 241

Hao Nguyen

https://youtu.be/r9PmRXQPhU0?t=115

https://youtu.be/lo0cp2uhxb0?t=57

Blue-Meth Synthesis

A possible alternative route is based on phenyl acetic acid and acetic acid, e.g., acetic acid anhydride, and leads initially to phenyl acetone through reduction with supported thorium oxide, from thorium nitrate, as catalyst in a tube furnace [11]. This synthesis route is probably that followed by Walter and Jesse and is very demanding, not least due to the preparation of radioactive thorium.

There then follows the reaction of phenyl acetone with methylamine and the subsequent reduction of the resulting *N*-methylimine. These two steps can take place as a multistep or one-pot reaction. Mercury aluminium amalgam can be used as catalyst for the reduction, as discussed by Walter and Jesse. Episode (IV-1), however, only shows how aluminium granulate is added to the reactor. Sodium amalgam or lithium aluminium hydride would also be possible catalysts [3].

As the reaction is not stereospecific, it yields the racemate (*S*)/(*R*)-*N*-methyl methamphetamine (Note: This synthesis step could also be carried out stereospecifically with the aid of stereoselective catalysts or, alternatively, a stereospecific separation of the product could take place before or after crystallization. Unfortunately, there is no indication of the method given in Breaking Bad). The resulting product is then separated from any remaining starting materials or by-products by steam distillation. This produces the clouds of steam that are often seen in the series. Finally, the oil obtained from the distillation has to be crystallized from an organic solvent, e.g. toluene. So it is all quite simple – maybe too simple?

References

[3] N. Stojanovska, S. Fu, M. Tahtouh, T. Kelly, A. Beavis, K. P. Kirkbride, *Forensic Sci. Int.* **2013**, 224, 8–26. DOI: 10.1016/j.forsciint.2012.10.040

[11] R. M. Herbst, R. H. Manske, *Methyl Benzyl Ketone*, *Organic Synthese* **1936**, *XVI,* 47.

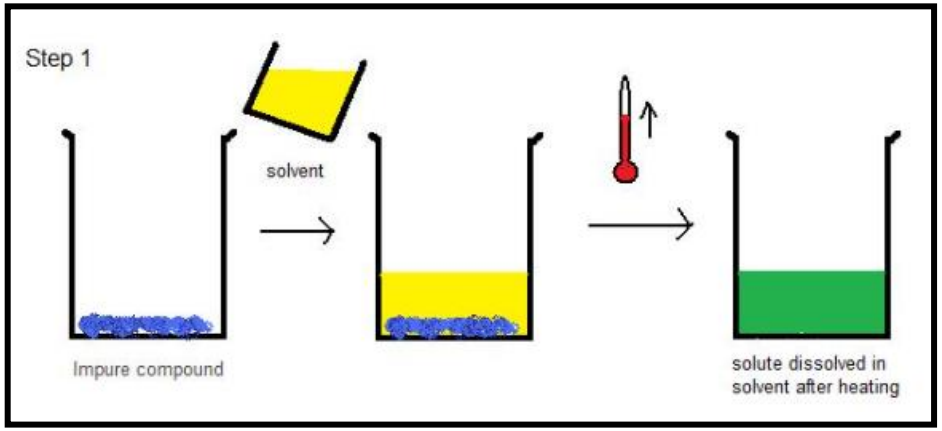
Recrystallizations

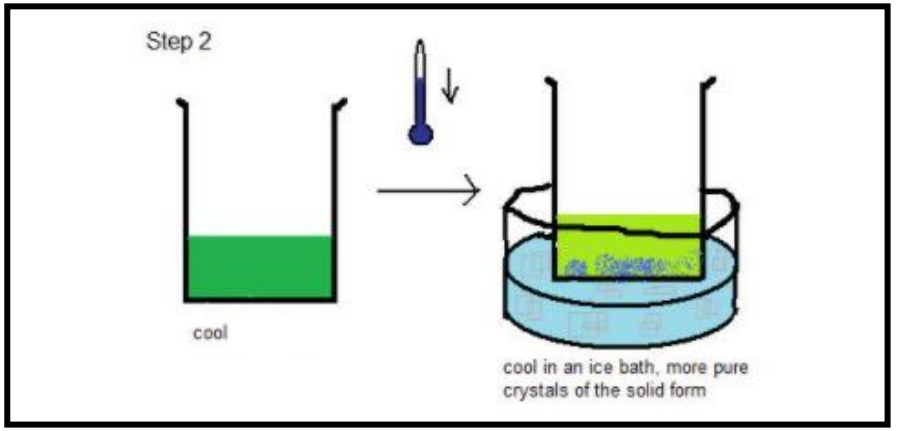
Purpose: Purify an impure compound using a solvent. Utilizes the principle that solubility is temperature dependent.

Overview: Dissolve crude crystals in the minimum amount of hot solvent to prepare a highly concentrated solution. Cool the solution. Since solubility decreases at lower temperatures, the product crystallizes while the impurities remain in solution.

Add just enough hot solvent to dissolve the solids.

Solvents





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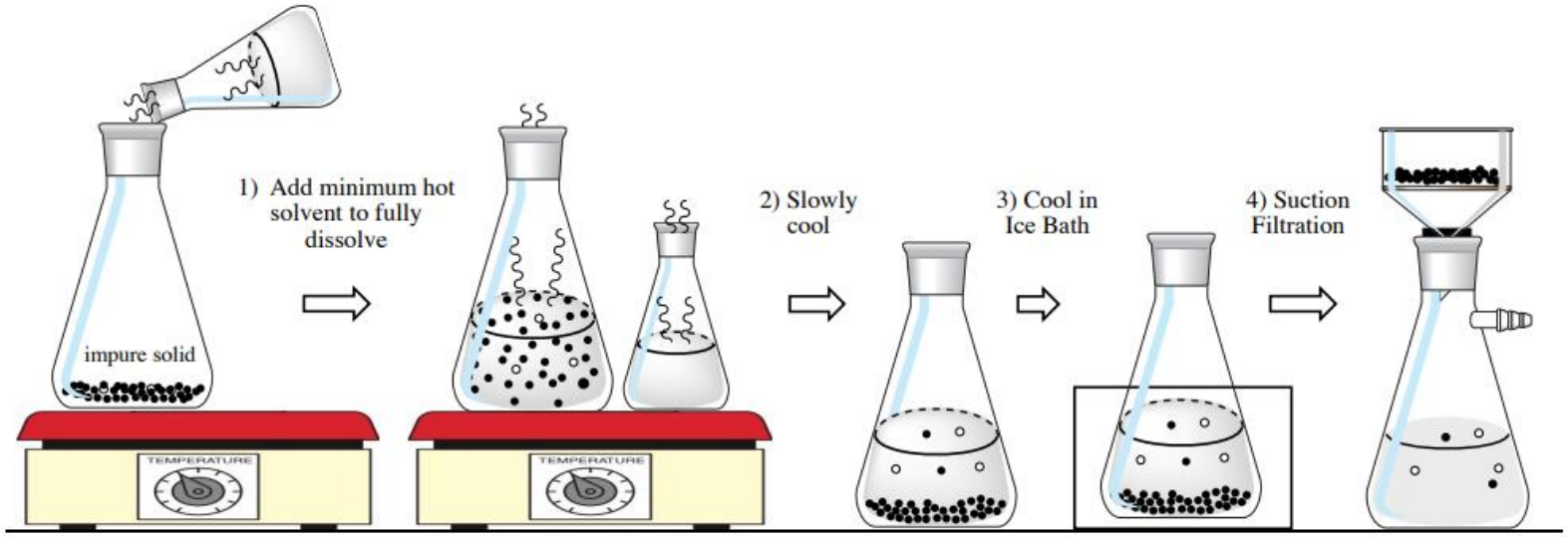
Recrystallizations: Procedure

- Heat solvent on hot plate in Erlenmeyer flask
- This may slowly start to boil away so you may have to refill
- Transfer small portion of hot solvent to crude product
- Place crude product mixture on hot plate and slowly add more hot solvent until all solids dissolve
    - Keep mixture on hot plate, if it cools solid will come out prematurely
    - Ensure solvent doesn't evaporate otherwise solids also come out and/or melt
- Once all solids dissolve, remove from hot plate and let cool to room temperature then place on ice

Recrystallizations

- Once solids form, filter to collect pure product
- If solids do not form scratch bottom of flask with metal spatula to initiate crystal growth
- Allow crystals to fully dry and then take melting point



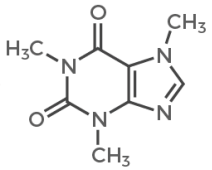
Extractions

THE CHEMISTRY OF DECAFFEINATED COFFEE

COFFEE & CAFFEINE

A typical cup of coffee contains between 70 to 140 milligrams of caffeine. Caffeine can influence the central nervous system, and can lead to sleep problems, restlessness and discomfort.

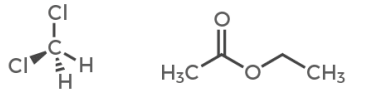
CAFFEINE



There are several processes used to decaffeinate coffee. In the U.S. decaffeination must remove 97% of the original caffeine content.

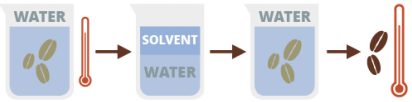
SOLVENT DECAFFEINATION

Solvent decaffeination uses solvents to selectively remove caffeine. Common solvents are methylene chloride and ethyl acetate.



Methylene chloride (left) and ethyl acetate (right)

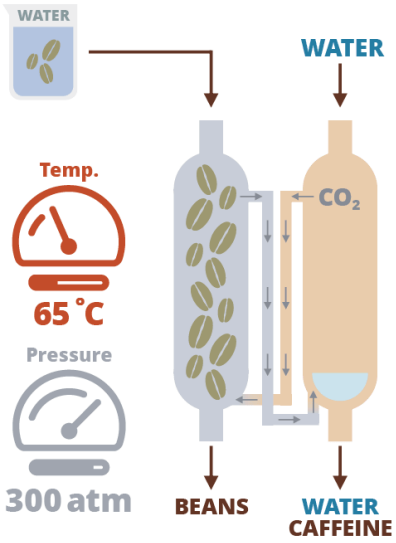
In direct solvent extraction (above) beans are steamed, then soaked in solvent to remove caffeine, before being steamed, dried, and roasted.



In indirect solvent extraction (above) beans are soaked in hot water. The water is then mixed with a solvent to remove caffeine, before flavours in the water are returned to the beans.

CO_2 DECAFFEINATION

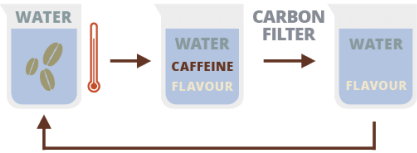
Solvent decaffeination removes caffeine but can also remove other flavour compounds or precursors. Carbon dioxide is more selective.



At high pressure carbon dioxide dissolves caffeine. The caffeine is removed from the carbon dioxide with water so it can be recirculated. The process lasts for up to 12 hours.

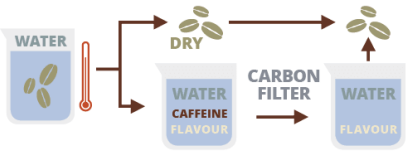
WATER DECAFFEINATION

'Swiss water decaffeination' soaks beans in hot water to remove caffeine and flavour compounds. Caffeine is removed from the water by filtration but flavour compounds remain. The flavour-saturated water removes caffeine from further bean batches without flavour loss.



Flavour-charged water used to remove caffeine from new batch of coffee beans

'French water decaffeination' soaks beans in hot water for 24 hours. The beans are removed and dried, and the water is filtered to remove caffeine. The caffeine-free water is added to the dried beans so that they reabsorb flavour compounds.



Extraction: Transfer of a compound from a solid or liquid into a different solvent or phase

Purpose: Separate compounds in a mixture. Often used as a purification step to isolate products in a reaction.

Sometimes referred to as reaction "work-up"

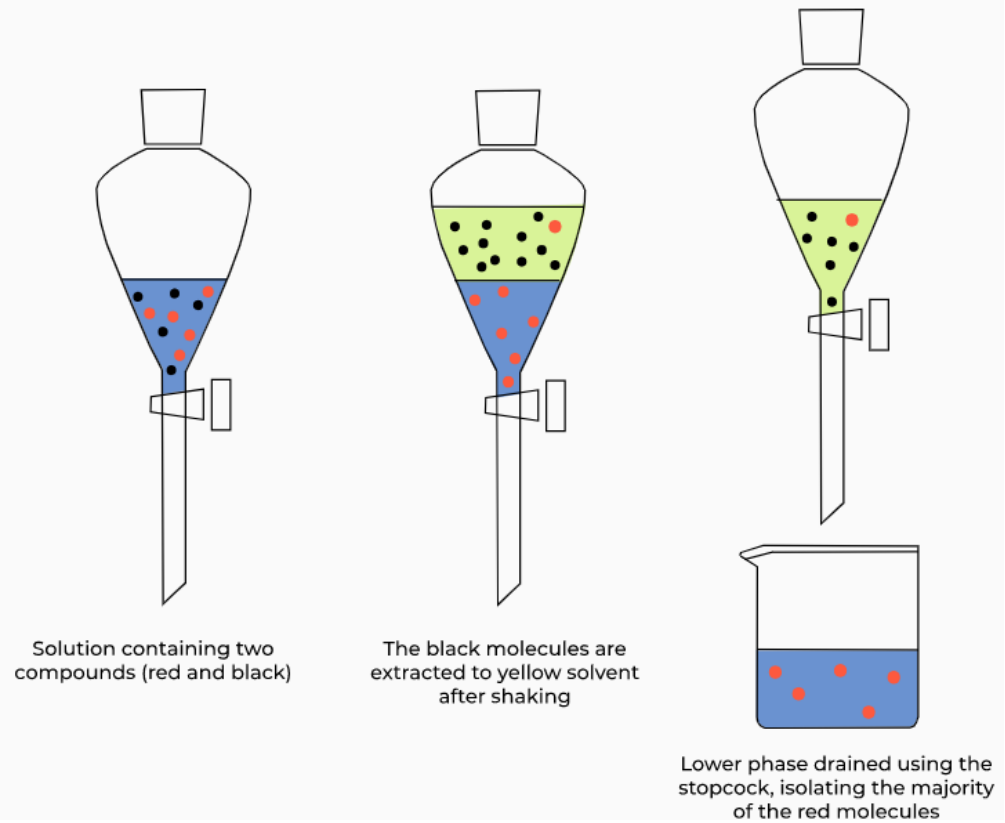
Overview: Use a separatory funnel containing two immiscible liquids, organic and aqueous. Depending on solubility, the compounds will dissolve in either the organic or aqueous layer.

- Examples of organic solvents: Diethyl ether (ether), dichloromethane/methylene chloride
- Examples of aqueous solutions: 1M HCl, 1M NaOH, saturated bicarbonate, Water, brine

Fundamentally based on density and solubility

Extractions

- After addition of solvents to separatory funnel, put in stop cock and shake vigorously
- Shaking allows for physical interactions between solvent particles and transfer of materials between layers
- DON'T FORGET TO VENT!!
  - Invert funnel every few shakes and open stopcock to relieve pressure

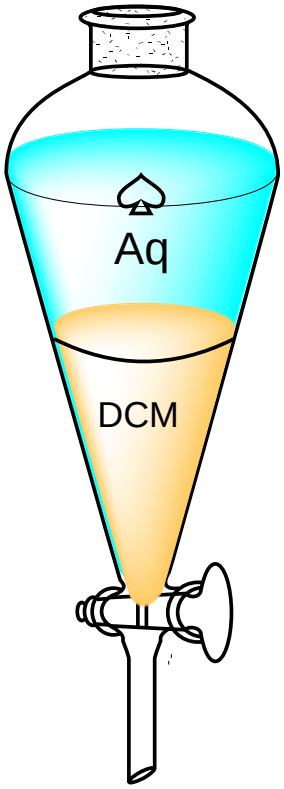


Extractions

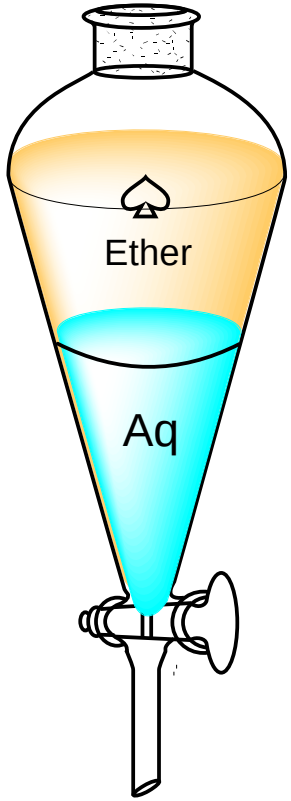
Density of solvents dictates which layer is on top

Methylene chloride (DCM) : 1.33 g/mL
Water (Aq): 1.00 g/mL
Diethyl ether (ether): 0.713 g/mL

DCM > Aq



Separatory Funnels



Ether < Aq

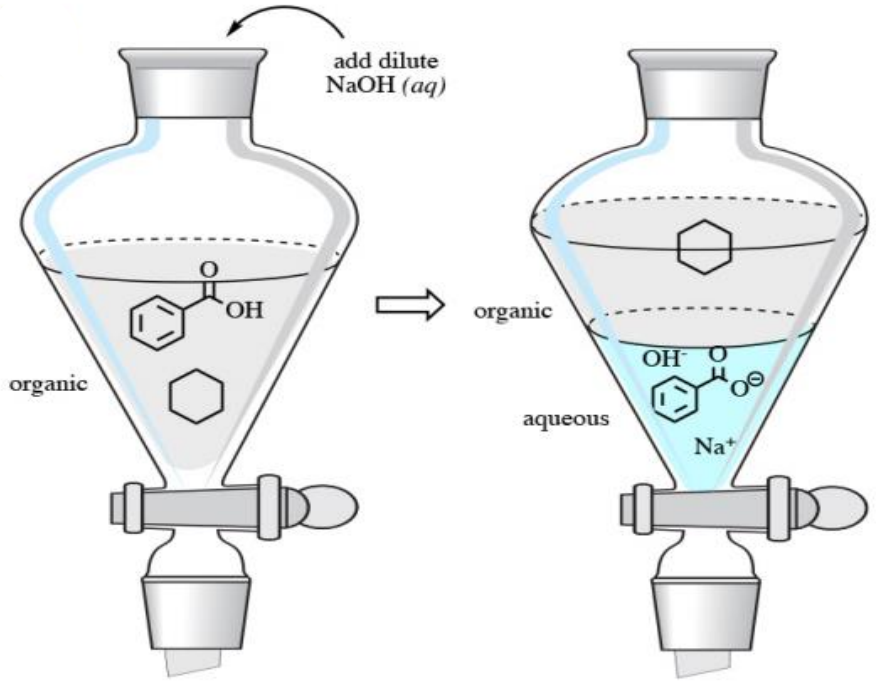
Extractions: aqueous washes

- NaOH: Deprotonates acids. If resulting compound is charged, increases solubility in water.
  - ie: carboxylic acid (organic) to carboxylate ion (aqueous)

- HCl: Protonates bases. If resulting compound is charged, increases solubility in water.

- Saturated $NaHCO_3$: neutralizes residual HCl or NaOH

- Brine: sequesters residual water, helps prevent or break up emulsions



DON'T throw any layers away until you are confident you have isolated your material

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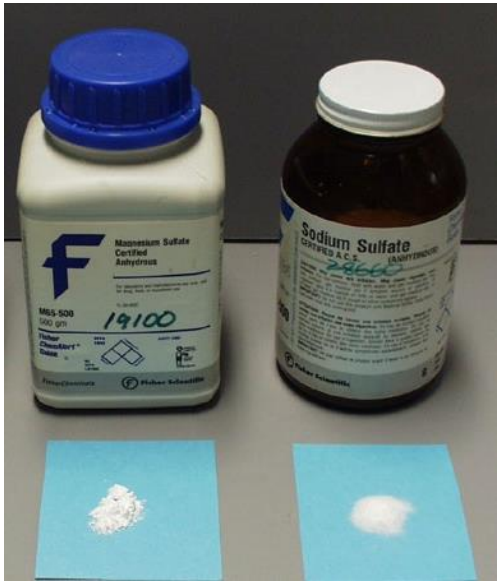
Extractions

Isolation of crude product

- Drain organic solvent from Separatory funnel into Erlenmeyer flask
- Add **drying agent** (Na_2SO_4 or $MgSO_4$) until it stops clumping and looks like snow globe
- Let sit for a few minutes
- Decant off solvent and evaporate with stream of air to provide crude product



Micro-scale Extractions