

Crystallization

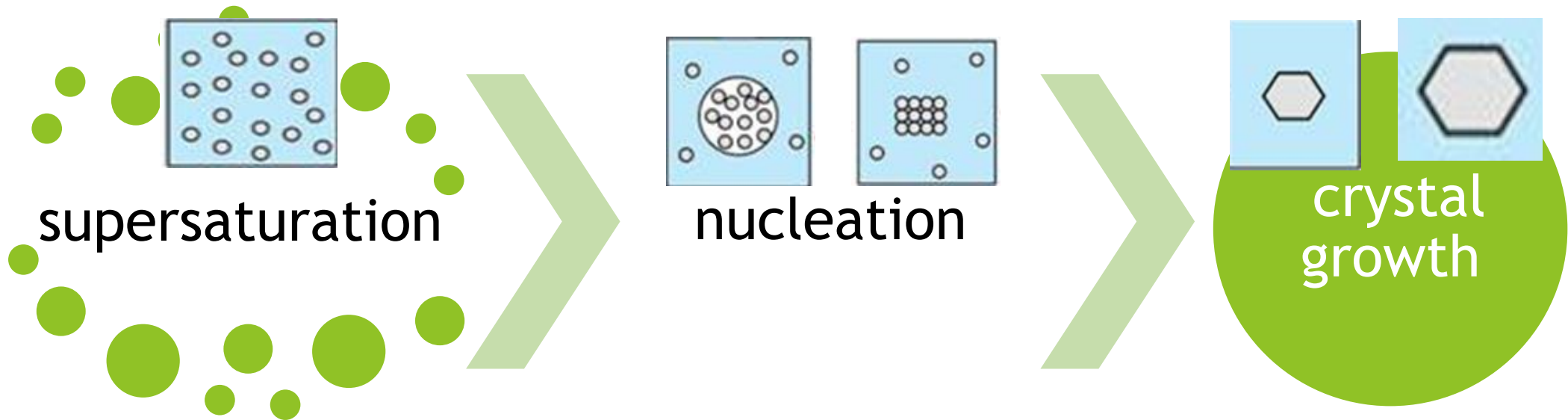
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Tinoco's Laboratory

Crystallization

- ▶ Process where a crystalline solid forms from a homogenous solution.



Steps



Uses

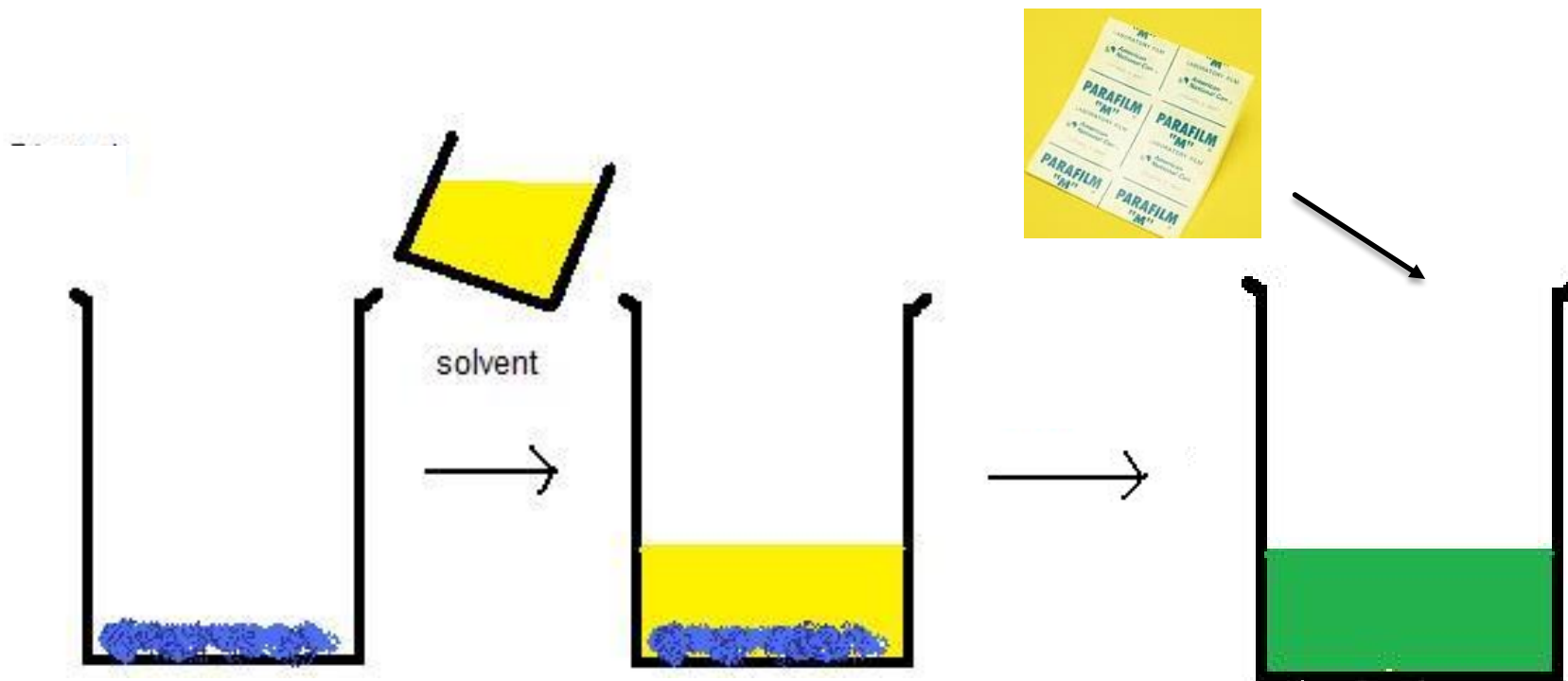
- ▶ Purification of a solid
- ▶ Obtaining solids for analysis (Example: X-Ray Diffraction)
- ▶ Improvement of physical and chemical stability of a compound

Methods

Considerations:

- ▶ Compound sensitivity (air, moisture, temperature)
- ▶ Amount of compound to be crystallized
- ▶ Vial/ flask/tube and surroundings where crystals will form must be clean
- ▶ Solvents

Slow evaporation



1) Make saturated or nearly saturated solution of compound that will be crystallized.

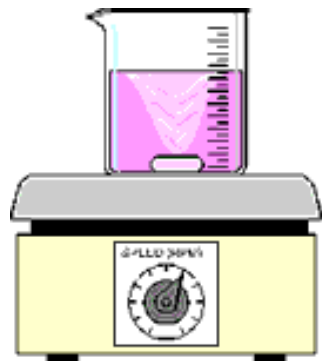
2) Seal with parafilm or aluminum foil and poke holes.

3) Leave in hood at room temperature until crystals form

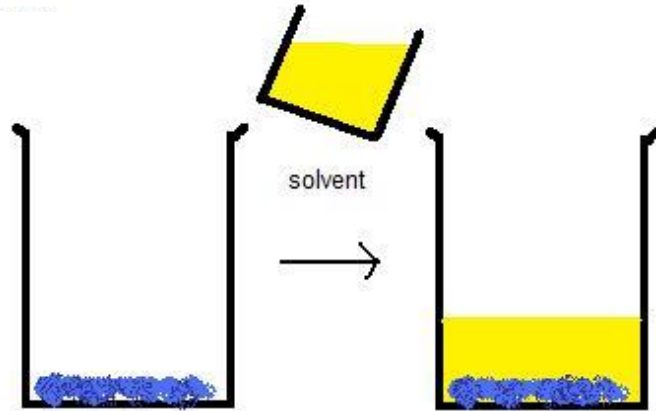
Libretexts. (2016, July 21) Recrystallization. Chemistry LibreTexts.

[https://chem.libretexts.org/Textbook_Maps/Physical_and_Theoretical_Chemistry_Textbook_Maps/Supplemental_Modules_\(Physical_and_Theoretical_Chemistry\)/Physical_Properties_of_Matter/Solutions_and_Mixtures/Case_Studies/RECRYSTALLIZATION](https://chem.libretexts.org/Textbook_Maps/Physical_and_Theoretical_Chemistry_Textbook_Maps/Supplemental_Modules_(Physical_and_Theoretical_Chemistry)/Physical_Properties_of_Matter/Solutions_and_Mixtures/Case_Studies/RECRYSTALLIZATION) (accessed October 2, 2018)

Slow Cooling



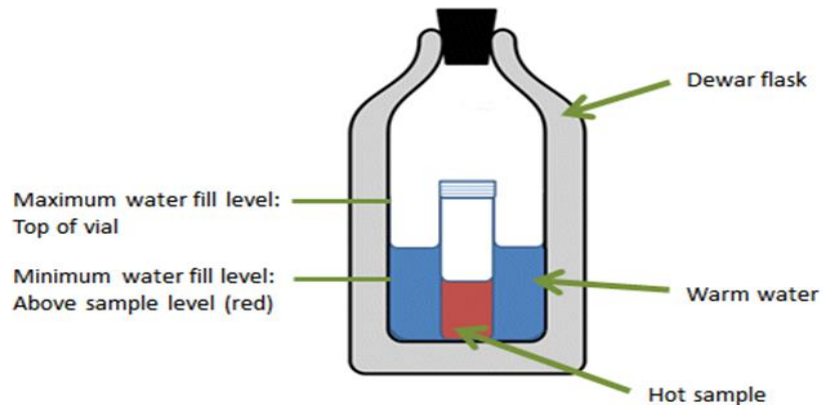
1) Heat solvent just to boiling point or some degrees below it.



2) Prepare a saturated solution.



3. Transfer solution to large test tube with stopper.

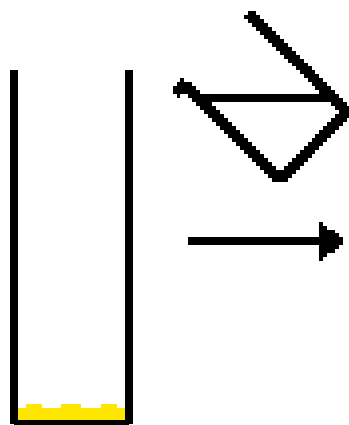


4) Transfer test tube into Dewar flask with hot water. Amount of water must exceed solvent level, but can't exceed test tube height.

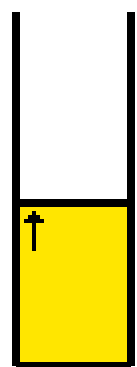
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5) Sit until solvent is cooled and crystals are formed. (1 week or more)

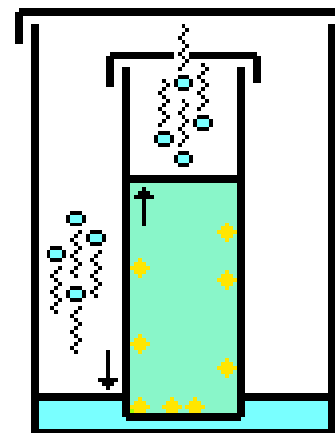
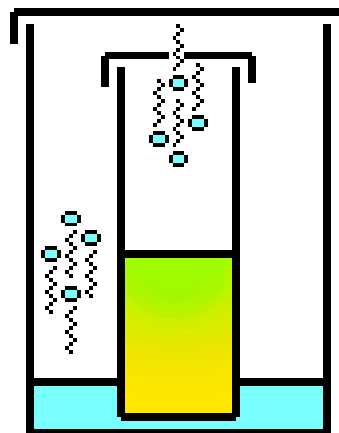
Vapor Diffusion



1. Prepare concentrated solution in vial.



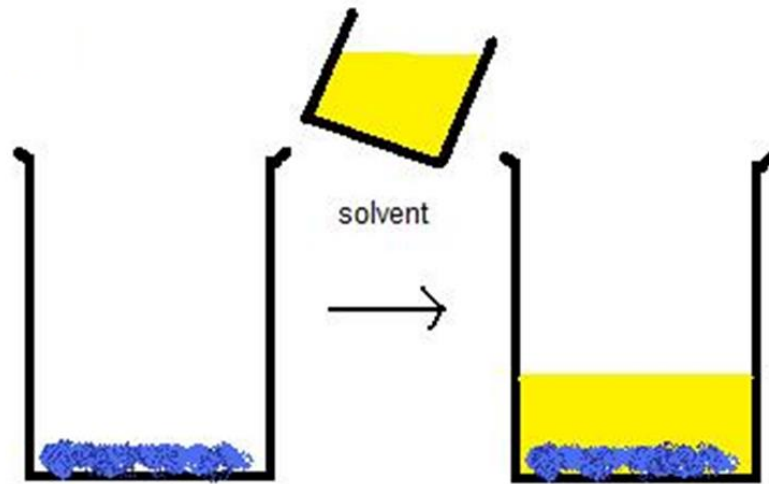
2. In a larger vial, add three equivalents of a second solvent. This second solvent must be miscible with the first solvent, and the compound that will be crystallized must be insoluble in it. Also, it must be more volatile than solvent 1. Then, put the first vial in this second vial.



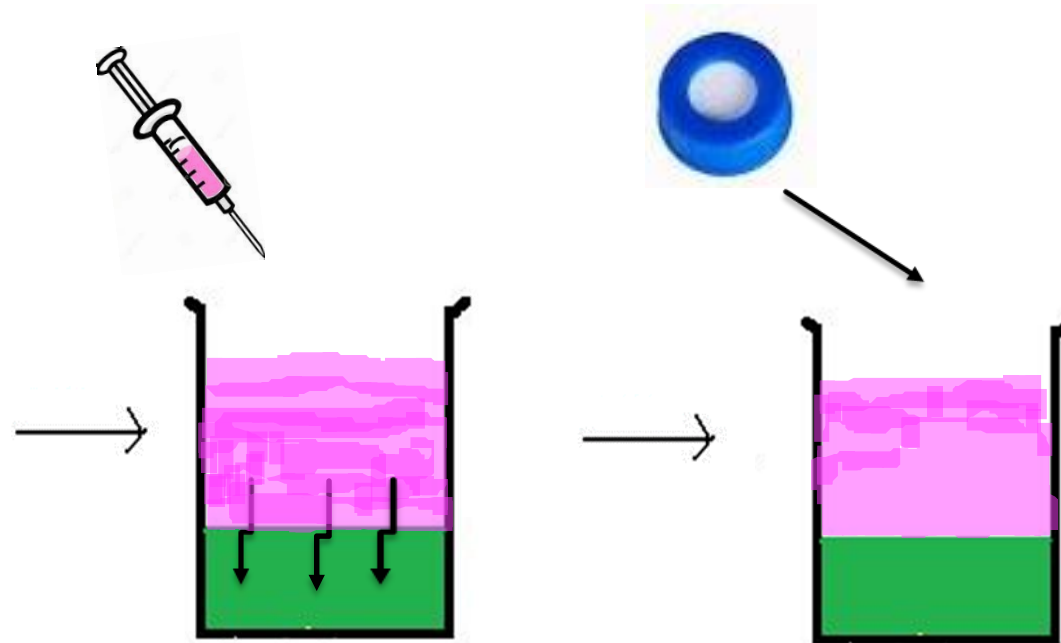
3) Close with cap and sit in a quiet place until crystals form.



Solvent Diffusion



1. Prepare a concentrated solution of compound to be crystallized in vial.



2. Carefully, add 1 equivalent of a second solvent, forming a clear and visible layer between each liquid. This second solvent must be miscible with the first solvent, but the compound that will be crystallized must be insoluble in it

3) Close with cap and sit in a quiet place (25 °C) until crystals form.

References

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Questions