



再結晶與熔點測定

Crystallization and Melting Point

<http://orglab.thu.edu.tw>



(1) Solubility: (a) $T \uparrow \rightarrow \text{solubility} \uparrow$
(b) like dissolves like
(functional group, polarity)

(2) Crystallization: Saturation at high temp.

↓ cool

Super-saturation at low temp.

↓

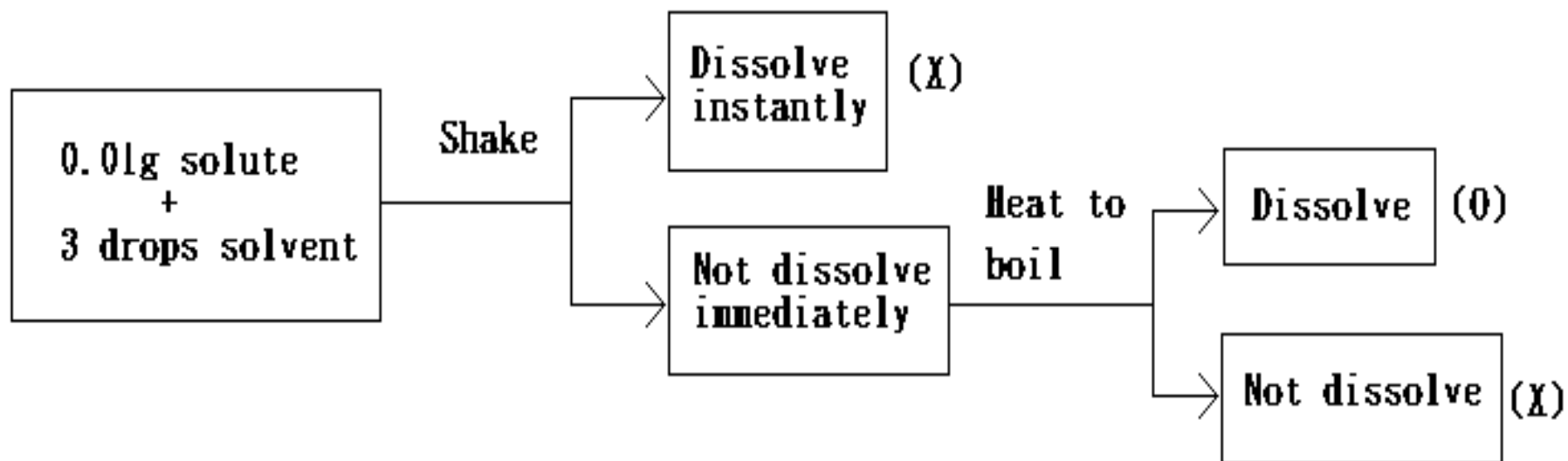
Crystallizing the solute



Choosing the Solvent

有機化學實驗

再結晶



Melting Point Measurement

有機化學實驗

再結晶

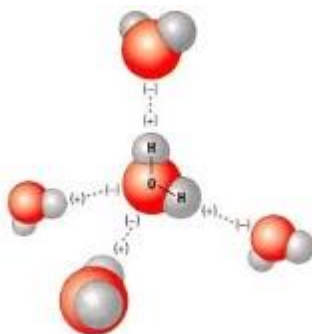
(1) A pure solid will melt reproducibly over a narrow range of temperature typically **less than 1°C**

→if the melting point does not rise after recrystallization

(2) Intermolecular force:

Ionic attraction

Hydrogen bonding



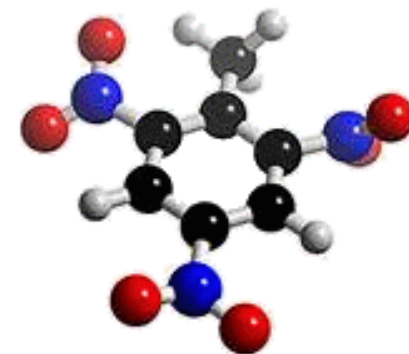
van der Waals force

Dipole-dipole interaction

Melting Point Measurement

有機化學實驗

再結晶



(3) Other factors :

(a) same functional group : M.W.↑ **Tm.p.↑**

(b) Symmetry↑ **Tm.p.↑**

(c) R.S enantiomer **same Tm.p.**

(d) with H.B. **Tm.p.↑** (under similar M.W.)

Recrystallization of Benzoic Acid

有機化學實驗

再結晶

0.5g benzoic acid + 15mL H₂O in a 50 mL Erlenmeyer flask



heat to **boil** on Hot Plate (~100°C)



to add H₂O (dropwise) until the benzoic acid just dissolves



add **hot** distilled H₂O (3~5 mL)



heat to boil for 10 sec (~100°C)



Gravity filtration of **hot** solution (apparatus)



evaporate the solution (**until volume ~15mL**)



Recrystallization of naphthalene

有機化學實驗

再結晶

- ↓
cool without being disturbed to room temp.
- ↓
collect with **suction filtration**
(if there are crystals on the flasks, use filtrate to rinse it)
- ↓
wash with the cold distilled H₂O
- ↓
dry
- ↓
weight (calculate the % yield)
- ↓
m.p. measurement



1. 繳交結晶產物,(結晶越**大** ,外形越漂亮 ,分數越高)。
2. 成品可拍照 , 於期末整理相簿**上傳 FB 實驗成果園地**:
需註明系級、組別實驗名稱。
3. 告訴助教回收**重量**與 m.p. , 回收率去再算。
4. 實驗問題 : **1 , 3**
5. 下次實驗「咖啡因萃取」**不做** , 只做酸鹼萃取。





Thank you !

<http://orglab.thu.edu.tw>

