

Solvents, Recrystallization, and Melting Point

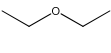


Intermolecular Forces




- Hydrogen bonding
 - Acceptor/donor
 - Dipole/dipole
 - Dipole/induced dipole
 - Induced dipole/induced dipole
 - Also called London dispersion forces
- VDWs Forces




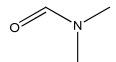

General Principles

- If molecules are about the same mass
 - H-bonding > Dipole-dipole > Induced dipole
- Induced dipole is weakest interaction, but as surface area increases, cumulative effect can overcome stronger IMF
- Strategy for predicting relative boiling points

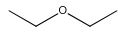


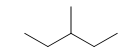
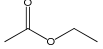




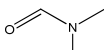

Solvents and Boiling Points

| | | | | |
|---|--------------------------------------|---|--------------------|---|
|  | CH ₂ Cl ₂ |  | CH ₃ OH |  |
| Ether (diethyl ether) | Dichloromethane (methylene chloride) | acetone | methanol | Tetrahydrofuran (THF) |
| 35 °C | 40 °C | 56 °C | 65 °C | 66 °C |

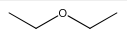


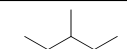
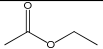




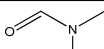

| | | | | |
|---|---|---|--------------------|------------------|
|  |  |  | CH ₃ CN | H ₂ O |
| hexanes | Ethyl acetate (EtOAc) | ethanol | acetonitrile | water |
| 69 °C | 77 °C | 78 °C | 82 °C | 100 °C |

| | | | | |
|---|---|---|---|---|
|  |  |  |  |  |
| toluene | Pyridine (pyr) | Acetic acid | Dimethyl formamide (DMF) | Dimethylsulfoxide (DMSO) |
| 111 °C | 115 °C | 118 °C | 153 °C | 189 °C |

H-Bond Donor, Acceptor, Neither, or Both

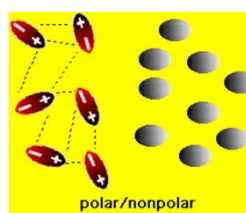
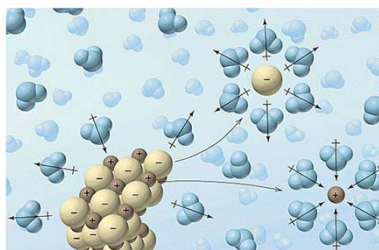
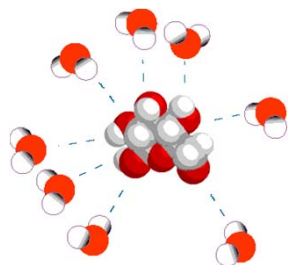
| | | | | |
|---|---|---|---|---|
|  | <chem>CH2Cl2</chem> |  | <chem>CH3OH</chem> |  |
| Ether (diethyl ether) | Dichloromethane (methylene chloride) | acetone | methanol | Tetrahydrofuran (THF) |
| 35 °C | 40 °C | 56 °C | 65 °C | 66 °C |
| | | | | |
|  |  |  | <chem>CH3CN</chem> | <chem>H2O</chem> |
| hexanes | Ethyl acetate (EtOAc) | ethanol | acetonitrile | water |
| 69 °C | 77 °C | 78 °C | 82 °C | 100 °C |
| | | | | |
|  |  |  |  |  |
| toluene | Pyridine (pyr) | Acetic acid | Dimethyl formamide (DMF) | Dimethylsulfoxide (DMSO) |
| 111 °C | 115 °C | 118 °C | 153 °C | 189 °C |
| | | | | |

Polar Protic, Polar Aprotic, Nonpolar

| | | | | |
|---|---|---|---|---|
|  | <chem>CH2Cl2</chem> |  | <chem>CH3OH</chem> |  |
| Ether (diethyl ether) | Dichloromethane (methylene chloride) | acetone | methanol | Tetrahydrofuran (THF) |
| 35 °C | 40 °C | 56 °C | 65 °C | 66 °C |
| | | | | |
|  |  |  | <chem>CH3CN</chem> | <chem>H2O</chem> |
| hexanes | Ethyl acetate (EtOAc) | ethanol | acetonitrile | water |
| 69 °C | 77 °C | 78 °C | 82 °C | 100 °C |
| | | | | |
|  |  |  |  |  |
| toluene | Pyridine (pyr) | Acetic acid | Dimethyl formamide (DMF) | Dimethylsulfoxide (DMSO) |
| 111 °C | 115 °C | 118 °C | 153 °C | 189 °C |
| | | | | |

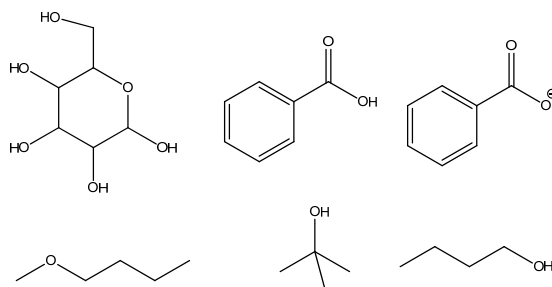
Solubility

- “Like dissolves like”
- Based on IMF



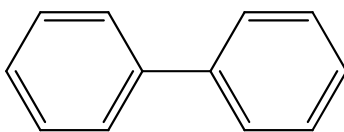
Solubility Rules of Thumb

- Water soluble if one H-bond donor per 3C
- Water soluble if ionic and less than 8C
- Water soluble if small, highly polar



Lab 2a: Purification of Biphenyl by Recrystallization

- **Question:** What solvent can be used for an effective recrystallization of biphenyl? How well does recrystallization work as a purification?



Applying Solvents and Intermolecular Forces

- Purification: separation of target compound from impurities
- Characterization
 - Identity: Do I have what I think I have?
 - Purity: How pure is the compound?

Recrystallization

- Purification for solids
- Useful for
 - Large samples
 - Final purification step
- Based on differential solubility



Practical Aspects

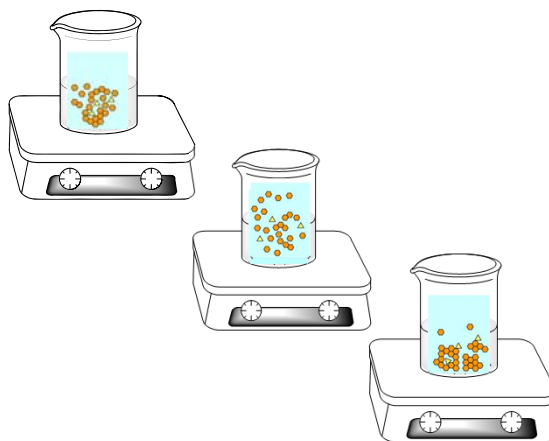
Impure Solid → Dissolve → Recrystallize → Filter



www.whfreeman.com/mohrig4e then watch the Chapter 15 video of recrystallization from mixed solvent

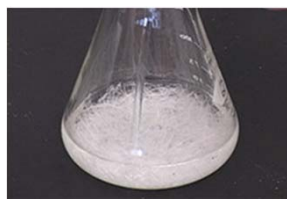
Recrystallization: Theory

- IMF in solids
- Temperature dependence
- Equilibrium process in slow crystal formation



Important Points

- Choice of solvent
- Mixed solvents
- Maintaining hot solvent
 - Safety when boiling
- Hot filtration
- Inducing crystallization
- Washing crystals
- Too much/too little solvent
- Oiling out



Melting Point

- Physical property to characterize substance
 - Identity
 - purity



capillary



Melt-temp

www.whfreeman.com/mohrig4e then watch the Chapter 14 video of packing a capillary tube for melting point

Theory and Application

- Theory
 - Pure crystals and impure crystals
 - Intermolecular Forces
 - Lattice energy
- Application
 - Depressed MP
 - Broad MP
 - Mixed MP

Eutectic mixture

