

# Techniques of laboratory sample handling

representative sample  $\Rightarrow$  analytical sample

Sample treatment before analyte(s) isolation:

Homogenization I – desintegration

grinding

milling

cutting

scraping

trituration

Homogenization II – as Homogenization I + additional steps

porosity – creating, increasing

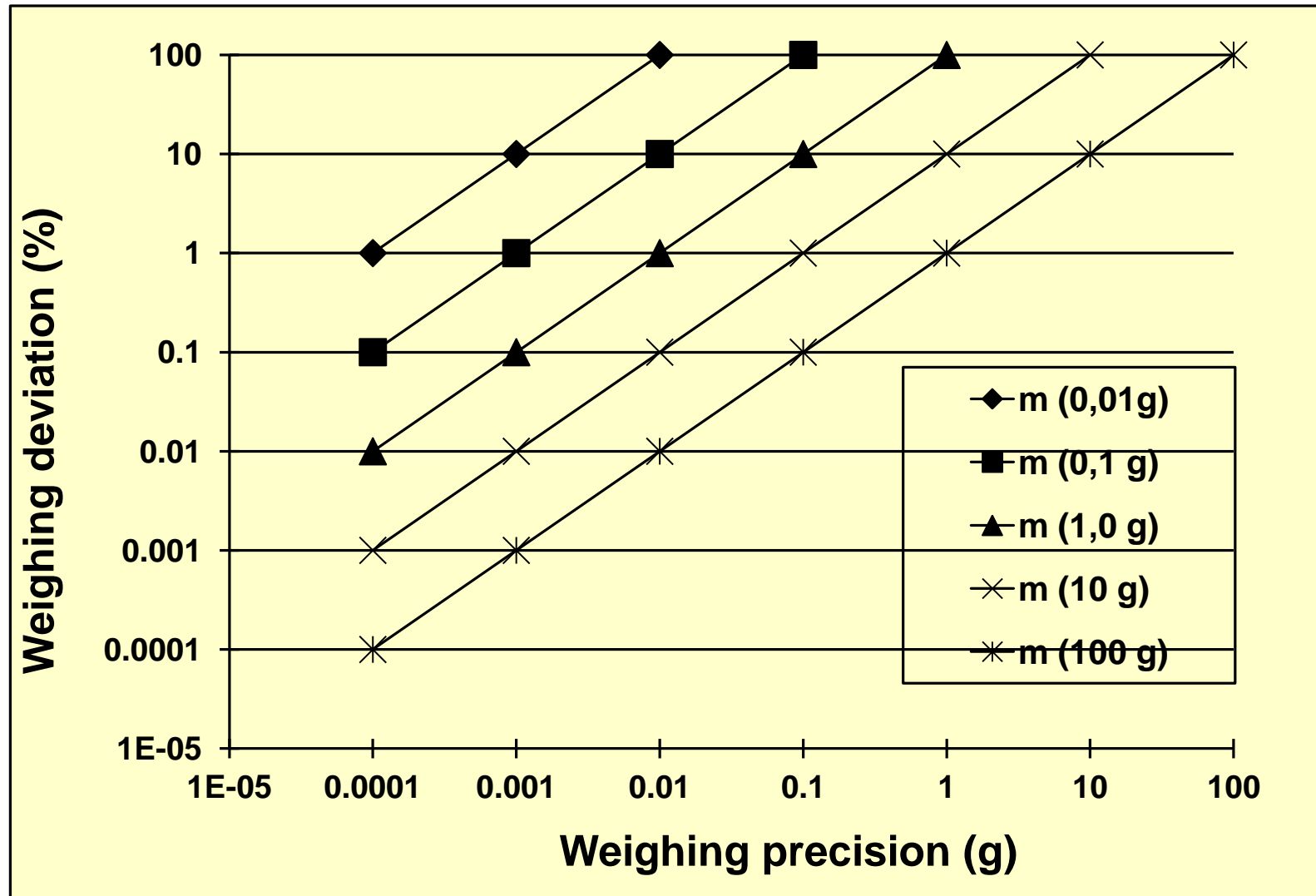
drying (water immobilization – crystalline form)

solvent addition or modification

pH adjustment



# Sample weighing – amount (adequacy – sufficiency), precision



**Analyte(s) isolation: transformation of analyte(s) to the form suitable for analysis**

***Characteristics of isolation procedure***

**Performance complexity: *isolation* - direct x indirect**

**Efficiency: % of isolated analytes (direct yield, recovery)**

**repeatability (concentration level)**

**Concentration: dilution or concentration**

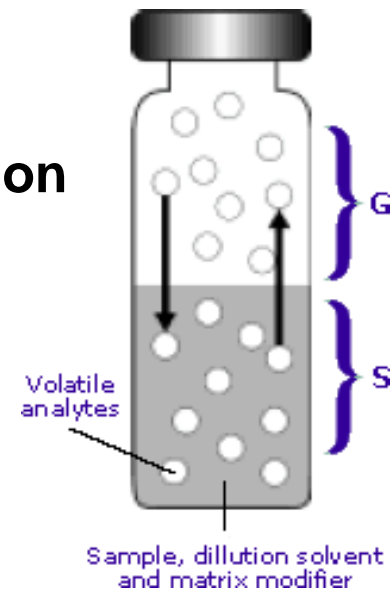
**Isolation medium phase: gas, liquid, solid, supercritical fluid**



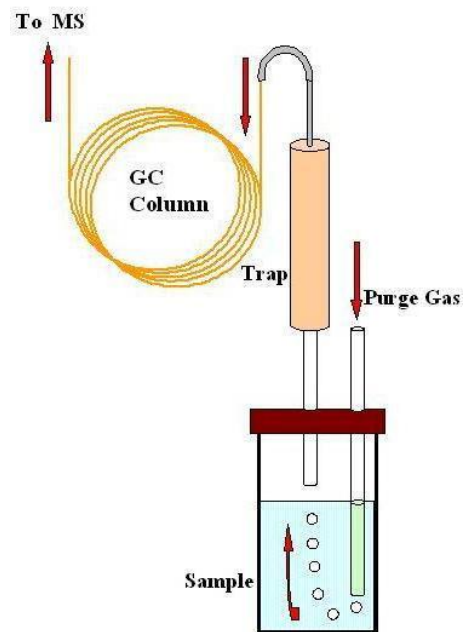
## Methods of isolation

### A) Gas Phase

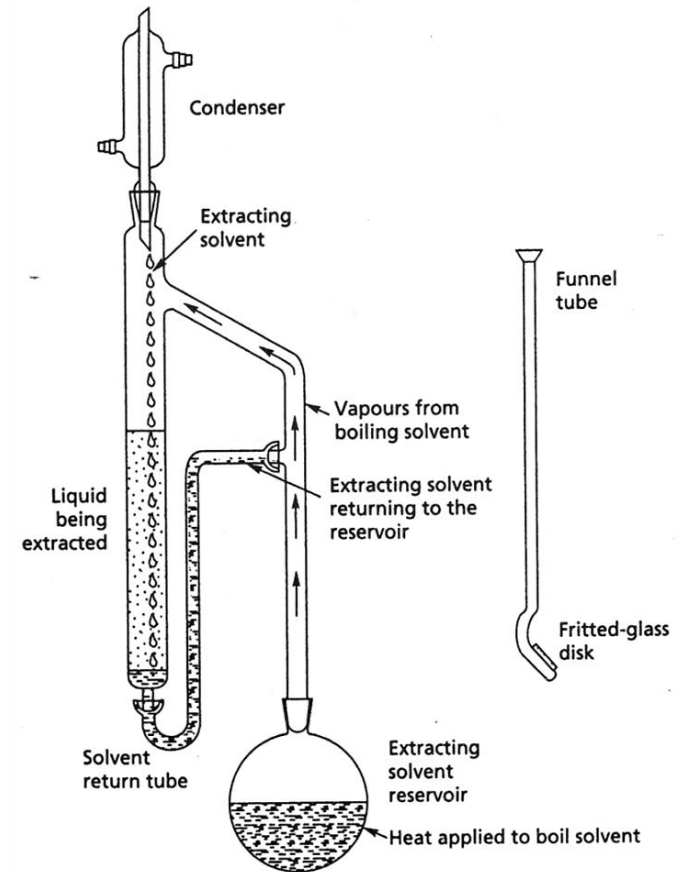
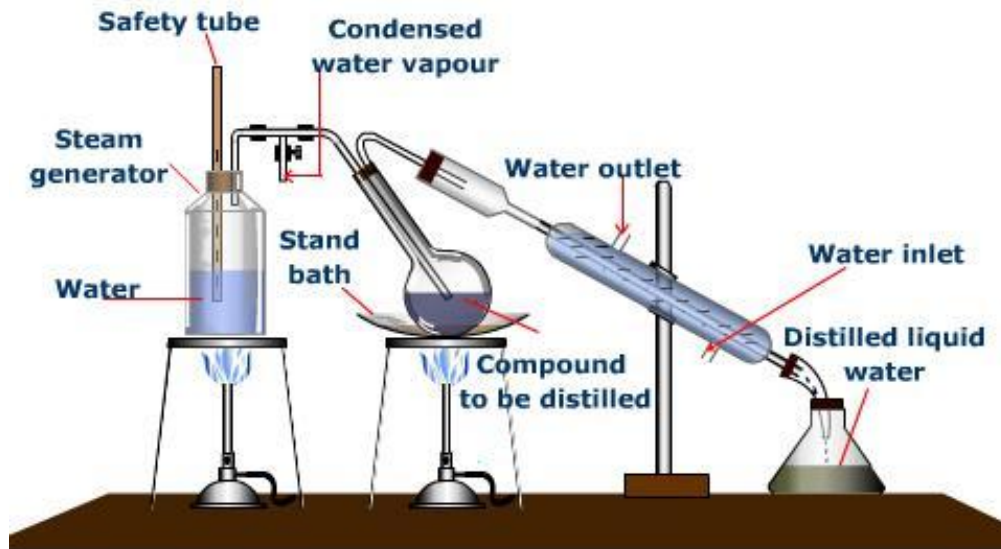
Head space (static, dynamic) - trap, cryofocustion



Purge & trap  
- trap, cryofocustion



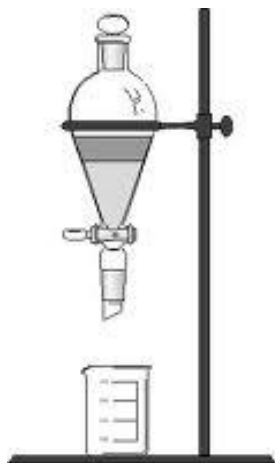
# Distillation - co-distillation with water steam or with other solvents



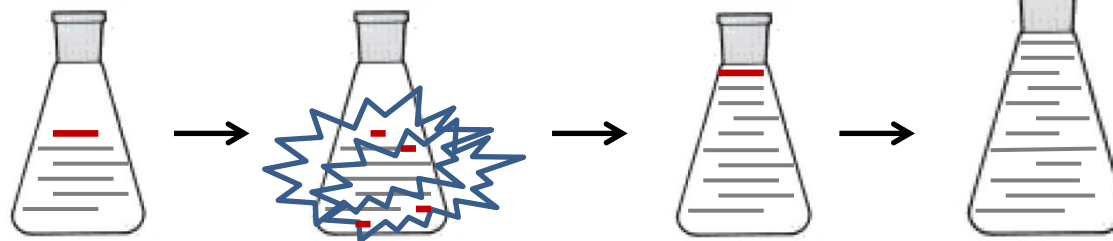
# Methods of isolation

## B) Liquid phase and SCF

### Liquid Liquid Extraction (LLE)



### Micro Extraction (ME) – micro LLE



# Methods of isolation

Liquid Solid Extraction (LSE)  
- agitation, sonication, reflux,  
Soxhlett, Soxtec, Twisselman

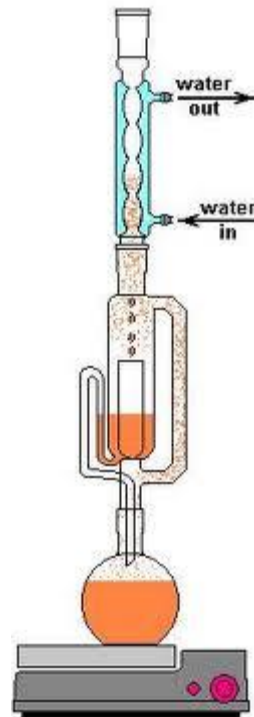


vibration

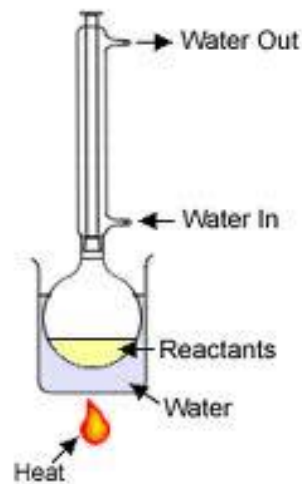
## Sonication



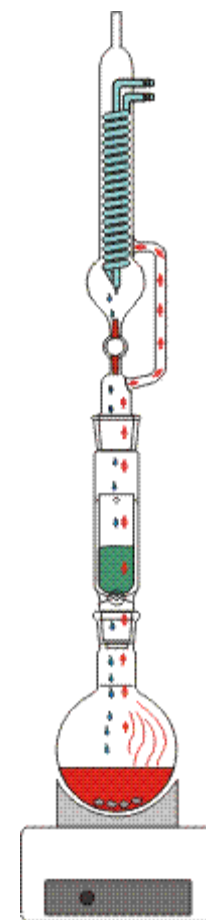
## Soxhlett



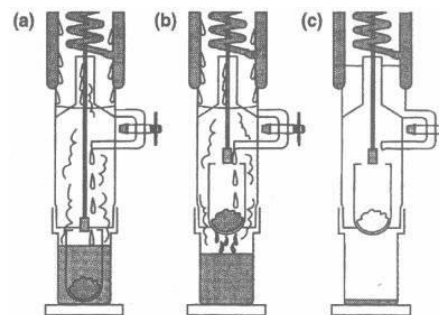
## Reflux



## Twisselman



## Soxtec

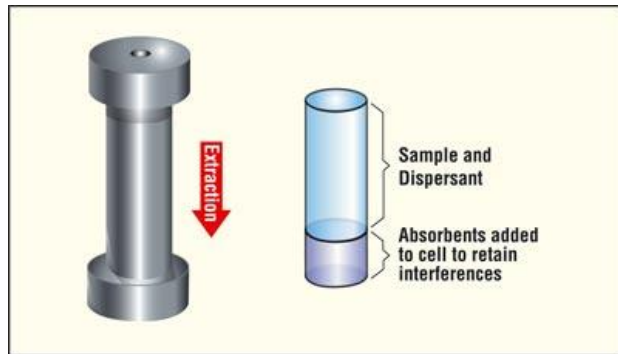


## Methods of isolation

### Microwave Assisted Solvent Extraction (MASE)



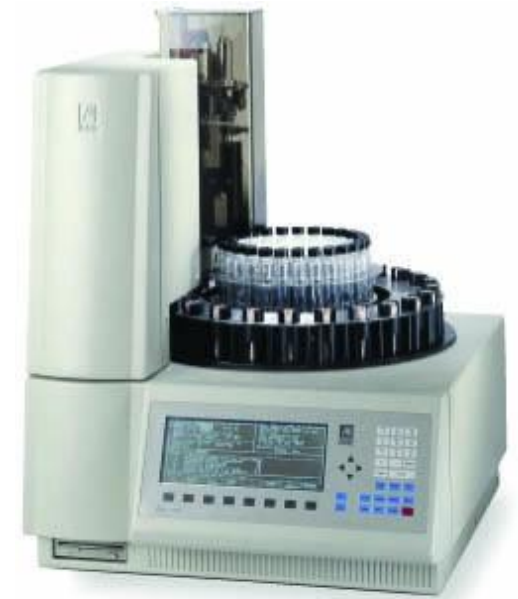
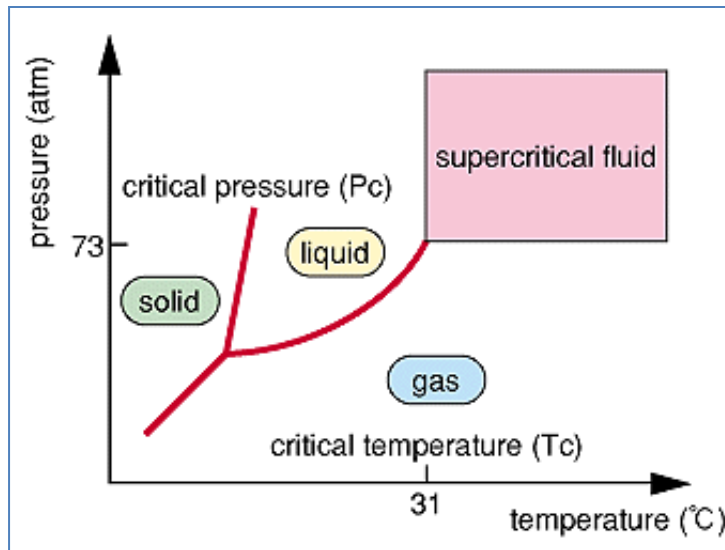
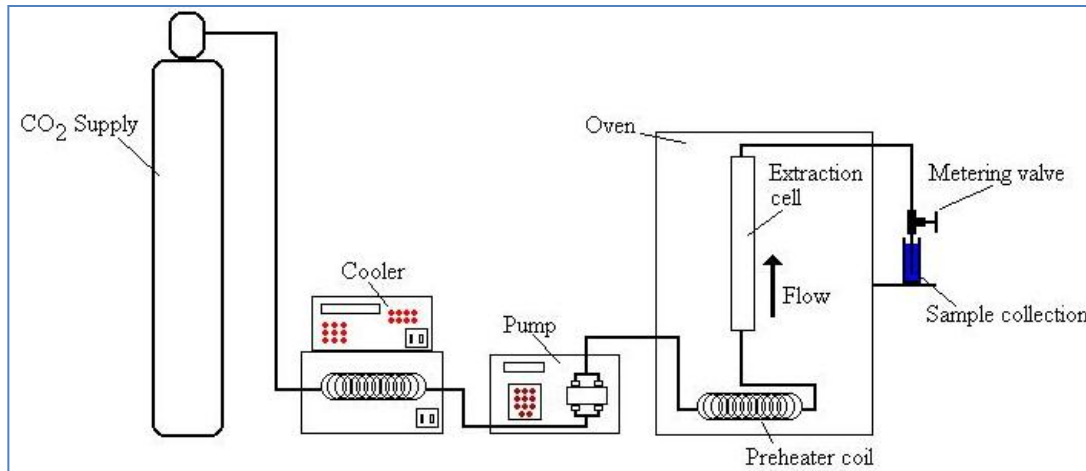
### Accelerated Solvent Extraction (ASE)





# Methods of isolation

## Supercritical Fluid Extraction (SFE)



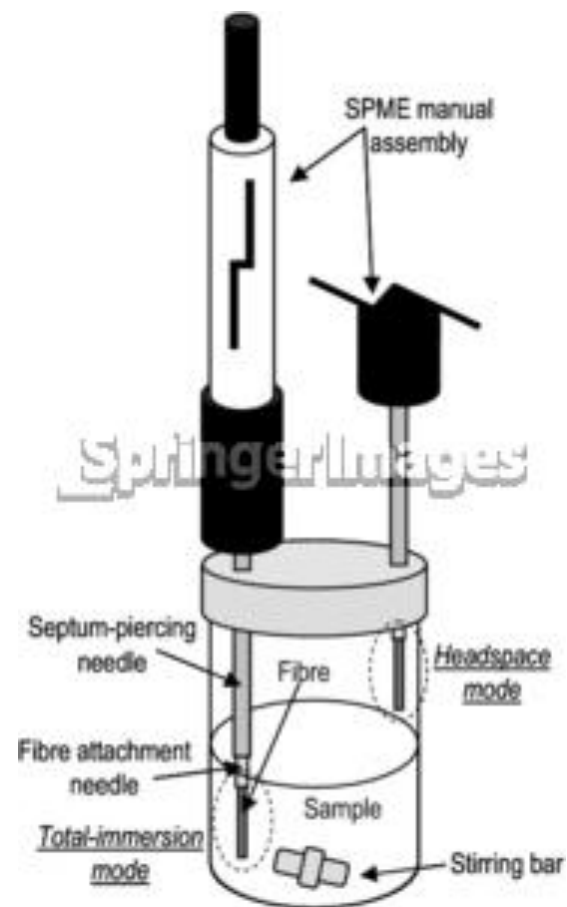
# Methods of isolation

## C) Solid phase

### Solid Phase Extraction (columns, discs) - SPE

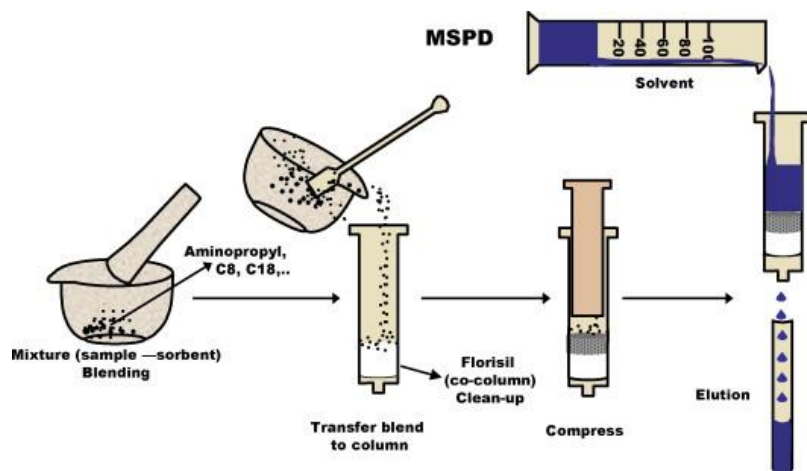


### Solid Phase Micro Extraction (SPME)

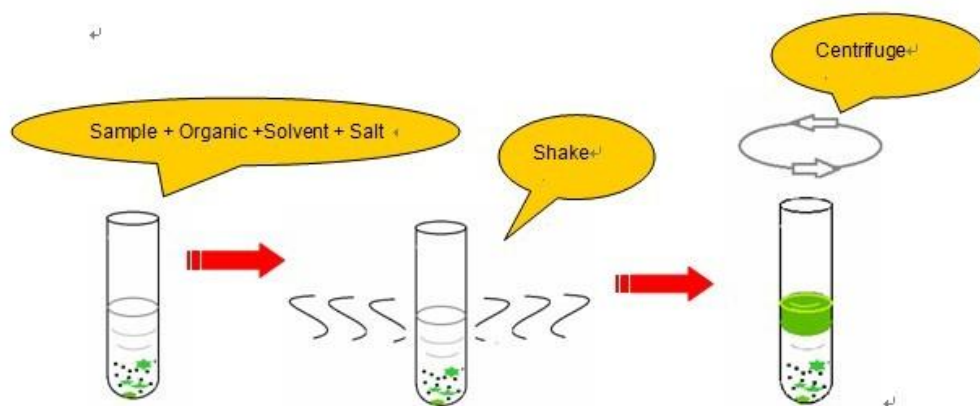


## Methods of isolation

### Matrix Solid Phase Dispersion (MSPD)



### Dispersive Solid Phase Extraction (d-SPE) (QuEChERS – Quick Easy Cheap Effective Rugged Safe)



## **Methods of isolation**

### **D) Decomposition**

**Mineralization – wet way X dry way**

**Supporting agents**

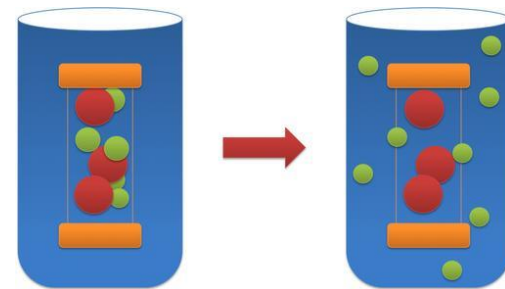
**Microwave heating**

**Microwave oven X focused microwaves**



## Preparative separation

Membrane separation - dialysis (microdialysis),  
static x dynamic



Precipitation (pH, salts, organic solvent, temperature)

Filtration



Extract drying, distillation, RVO,  
Kuderna-Danish, Snyder's column

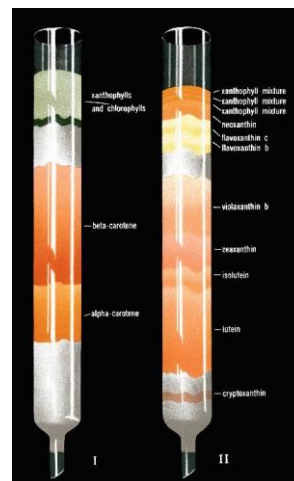
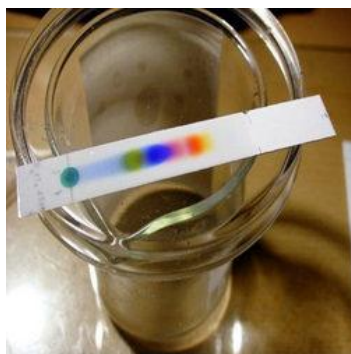
Centrifugation



## Preparative separation

LSC - Adsorption chromatography

TLC (HPTLC)



LC (HPLC) – elution, ionex, chiral etc.

Electromigration preparative separation

(Bio) Affinity methods (MSPD, SPE, LC – enzymatic, immunochemical)

GPC (HPGPC)

