Advanced Methods for Chemistry
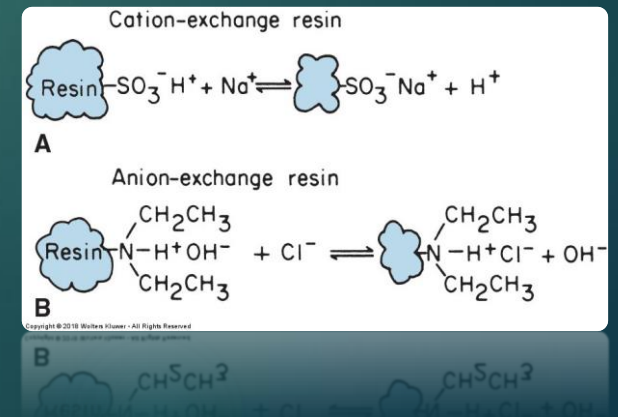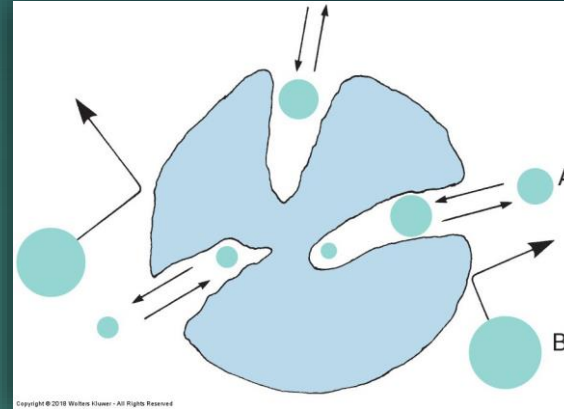
CHROMATOGRAPHY AND MASS-SPEC

Chromatography Methods

- Does not do the analysis itself
  - Requires detection method
- Separation of mixtures of substances
  - Adsorption
  - Partition
    - Normal
    - Reverse
  - Steric exclusion
  - Ion Exchange
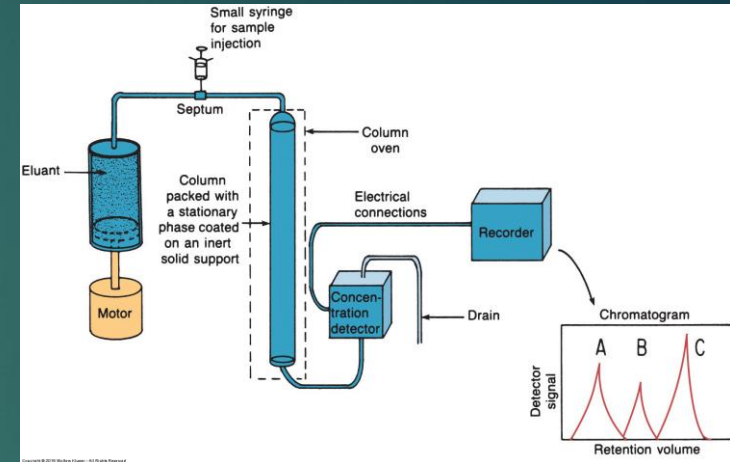
Chromatography Applications

- **Thin Layer Chromatography**
  - $R_f = \dfrac{distance\ of\ component}{distance\ of\ solvent}$
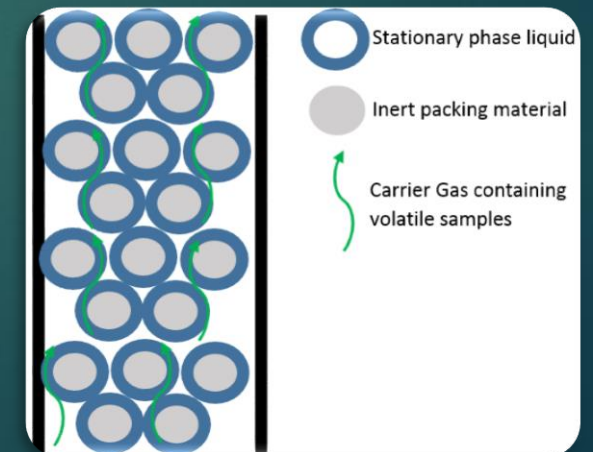- **High Performance Liquid Chromatography**
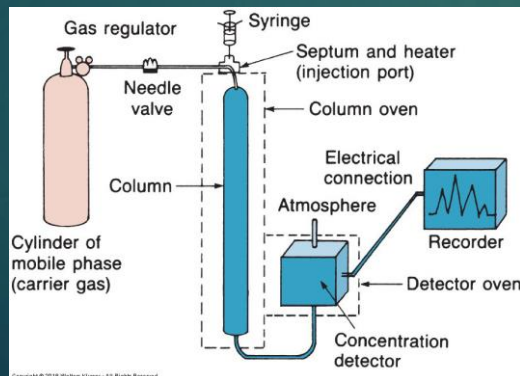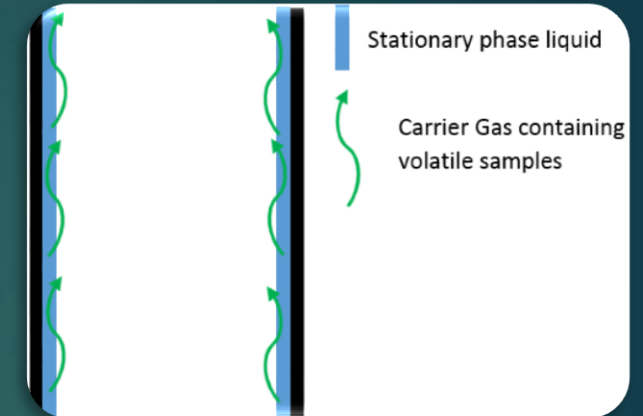 - Fine packing of column (usually polar)
  - Reverse phase uses non-polar stationary phase
  - Detection: spectrophotometric, amperometric, electrochemical, or MS
    - Internal standard used to standardize injection
  - Concentration determined by AUC (area under curve)
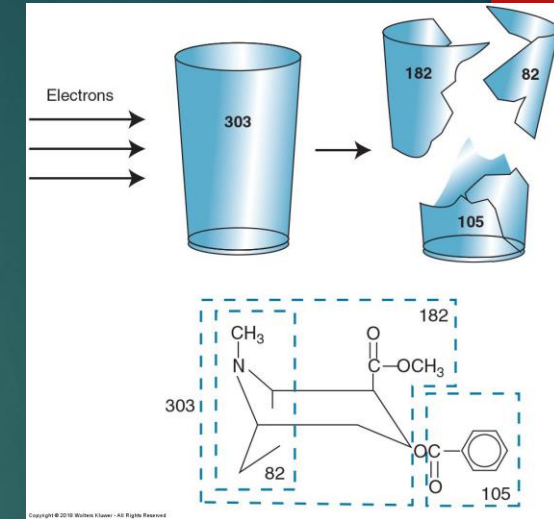
Chromatography Applications

- Gas Chromatography
  - Separation of volatile mixtures
  - Mobile phase gas, stationary phase usually a non-volatile, non-reactive liquid coated onto inert packing, or walls
    - May be non-selective (only volatility changes retention time)
    - May be selective (liquid phase also separates based on relative polarity)
  - Detectors: thermal conductivity, flame ionization

Stationary phase liquid

Carrier Gas containing volatile samples

Gas regulator
Syringe
Septum and heater (injection port)
Needle valve
Column oven
Column
Electrical connection
Atmosphere
Recorder
Cylinder of mobile phase (carrier gas)
Detector oven
Concentration detector

Stationary phase liquid

Inert packing material
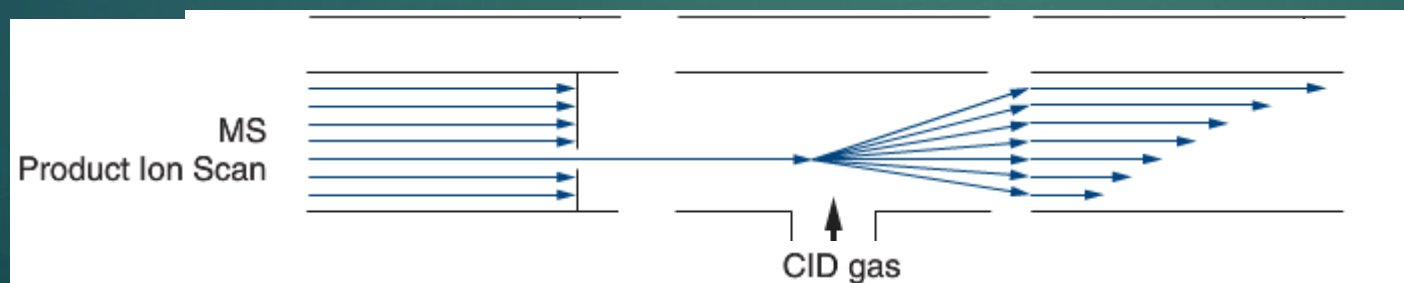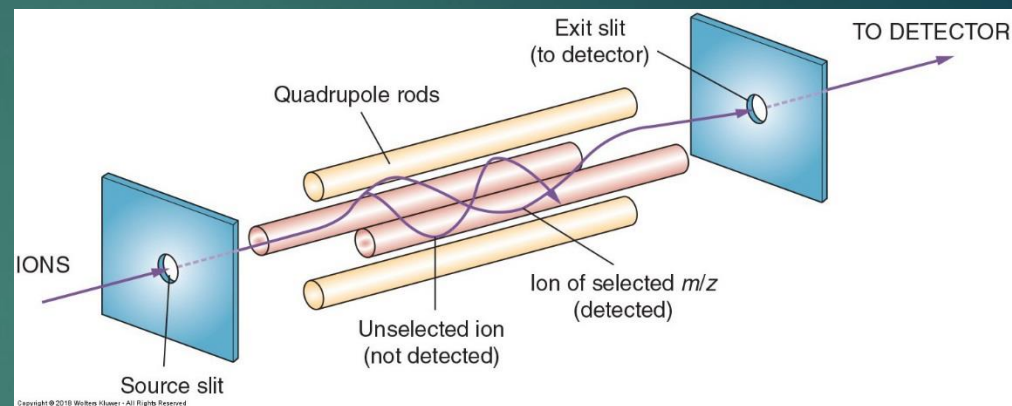
Carrier Gas containing volatile samples

Mass Spectrometry

▶ Detection of fragments based on m/z

  ▶ Ionization Methods

   ▶ Electron Ionization: shoot molecules with electrons

   ▶ Electrospray: sprayed through charged needle tip

   ▶ Chemical Ionization: APCI or regular CI

   ▶ Inductively Coupled Plasma: total ionization to atoms

Mass Spectrometry

- ▶ Separation Method
  - ▶ Quadrupoles
  - ▶ Quadrupoles in series serve more functions
  - ▶ Ion traps: modified quadrupoles build up ions of interest to increase sensitivity

Mass Spec Applications

- Detection after chromatography
  - HPLC
  - LC
  - GC
- Independent of previous method
  - ICP-MS
    - Replaces AAS
  - MS/MS