Analytical Techniques

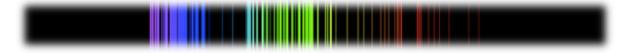
Atomic Absorption Spectroscopy

- Qualitative and Quantitative
- Metals- hollow metal cathode lamp for light (unique wavelength)
- Vaporised in a reducing flame- detects in gas form
- Valence electrons absorb wavelength of light (radiation) and jump to higher energy level
- Concentration determined by how much light absorbed- calibration curve

Atomic vapour Pulsed light Light Source Monochromator

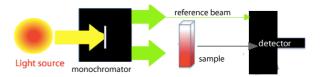
Atomic Emission Spectroscopy

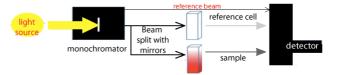
- Qualitative and Quantitative
- When electron returns to ground state, light is emitted
- Sample identified by wavelength of light emitted (from emission spectrum)
- Concentration identified by intensity of emitted light



UV Visible Spectroscopy

- Qualitative and Quantitative (mostly)
- Organic molecules and transition metals
- Process
 - Valence electrons absorb electromagnetic radiation in UV-Visible region
 - Measures light energy absorbed by electron as it jumps to excited state
 - Send in small band of wavelengths
 - Not as accurate as AAS- other things (glassware; solvent) absorb light
 - Need dilute solutions for light to pass through (not opaque like milk)
- If blue light seen; means red light absorbed
- Calibration curve- Absorbance vs. Concentration





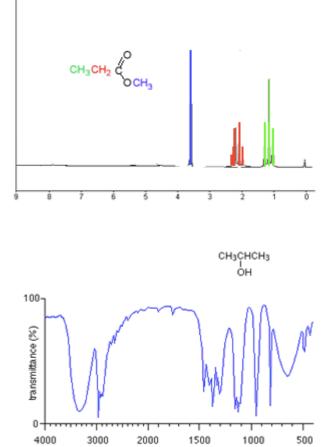
wavenumber (cm⁻¹)

NMR

- Qualitative (very expensive)
- Organic molecules
- For atoms with odd number of nucleons ¹HNMR and ¹³CNMR
- Excitation of nucleons to higher energy state using radio waves
- n+1 rule
- Closer to electronegative end (OH), further left it is on spectrum
- Hydrogen environments
- All the H on the neighbouring C
- TMS

Infrared Spectroscopy

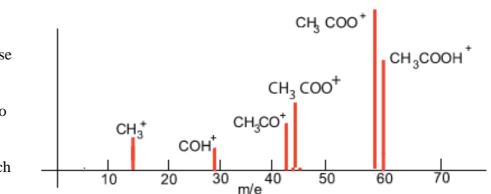
- Qualitative
- Absorption of infrared radiation by bonds
- Wavelength absorbed depends on the mass of atoms on either side and strength of bond
- Use Data Booklet



Mass Spectroscopy

- Qualitative and Quantitative
- Highly destructive technique- bombard molecule with high energy electrons
- Creates positive fragments- deflected onto detector plate
- m/z is mass/chargealways +
- If no charge, it passes straight through
- Molecular Mass of parent ion furthest peak on right (can lose 1 e⁻ and still stay together)
- May be isotope if two peaks close together
- Height of peak = % abundance- how much there is

Simplified mass spectrum of ethanoic acid



Column Chromatography

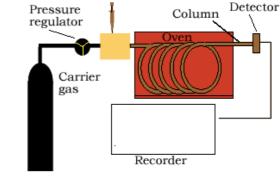
- Solid stationary phase- coated in viscous liquid
- Normally- Non-Polar mobile phase; Polar stationary phase
 - Otherwise, it is reversed-phase chromatography
- Continually **adsorb** (onto stationary) and **desorb** (into mobile)
- Rate of Flow (retention time)
 - How much compounds attracted to stationary (stick on)
 - How soluble it is in mobile phasesoluble it goes quicker- polarity
 - Like attracts Like

High Pressure Liquid Chromatography

- Solid phase from smaller particles
 - Increases surface area; more interaction (adsorption and desorption)
 - o Better separation
 - Slows down rate of flow through the column, and therefore high pressure used
- Uses UV light to detect
- Components identified by Retention Time; area under peak relative to concentration

Gas-Liquid Chromatography

- Inert gas is mobile phase (He)
- Liquid stationary phase (generally non-polar)
- Conditions:
 - Very sensitive- small amounts
 - Small compounds need to be vaporised without decomposing
 - \circ Pushed through column by gas
- Retention Time
 - Boiling point- high boiling point will spend more time as liquid at start- take longer to get through (larger molecules have higher boiling points)
 - Solubility in liquid phase- more soluble means stuck there longer- polarity



- Higher temp makes everything faster, especially smaller molecules- kinetic energy- affects separation
- Area under peak relative to concentration

