

Topic 11: Measurement and data processing

11.1 Uncertainties and errors in measurements and results

Quantitative and Qualitative Data

- Quantitative data is data **taken from measurements** made in the laboratory and is associated with random errors
- Qualitative data includes non-numerical data **obtained from observations**, not from measurements
- A results table should include quantitative data with units and uncertainties
- Quantitative data should be recorded to the appropriate precision
- Qualitative data should also be recorded

Absolute and Percentage uncertainties

- The absolute uncertainty of digital apparatus is \pm the smallest scale division

$$\text{Percentage Uncertainty} = \frac{\text{Absolute uncertainty}}{\text{Measurement}} \times 100\%$$

- Absolute uncertainty of mass balance is $\pm 0.01\text{g}$
- Absolute uncertainty of analog apparatus is \pm half the smallest scale division
 - Absolute uncertainty of measuring cylinder is $\pm 0.5\text{cm}^3$
- Absolute uncertainty of burette is $\pm 0.05\text{cm}^3$

Random Errors

- Random errors are caused by unpredictable changes in the experiment (in the conditions or apparatus)**
- With random errors, there is an equal probability of the measured value being too high or too low
- Examples of random error:
 - Changes in the environment during the experiment (such as a change in the room temperature)
 - Observer misinterpreting the reading
 - Insufficient data (not conducting repeat trials)
- Random errors cannot be eliminated but can be reduced by conducting repeat trials**
- They can also be reduced by using precise apparatus (such as a volumetric pipette rather than a beaker to measure volume)

Systematic Errors

- Systematic errors occur as a result of a flaw in the experimental design of apparatus
- Systematic errors cause the measured value to be consistently higher or lower than the actual value
- They cannot be reduced by conducting repeat trials
- Examples of systematic error:
 - Heat loss in an experiment to measure enthalpy change
 - Losing a product (such as a gas) in a reaction
 - Overshooting the endpoint in a titration
 - Reading from the top of the meniscus when measuring volume
 - Forgetting to zero a mass balance

Percentage error

- Percentage error is a measure of how close the experimental value is to the theoretical or accepted value

$$\text{Percentage Error} = \frac{\text{Experimental value} - \text{Theoretical value}}{\text{Theoretical value}} \times 100\%$$

- If the experimental value is less than the theoretical value, the percentage error will be negative

11.3 Spectroscopic identification of organic compounds

Index of Hydrogen Deficiency (IHD)

- The index of hydrogen deficiency (IHD) is a count of how many molecules of H₂ need to be added to convert the molecule to the corresponding, saturated, non-cyclic molecule. In other words a degree of unsaturation
- The IHD for a hydrocarbon with x carbon atoms and y hydrogen atoms:

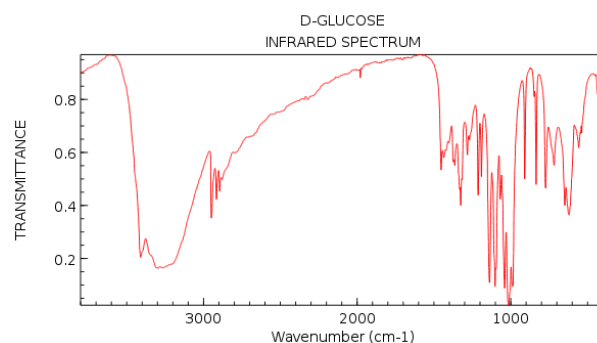
$$IHD = \frac{(2x + 2 - y)}{2} \times 100\%$$

- Note:
 - Sulfur and oxygen do not affect IHD
 - Halogens (F, Cl, Br and I) are treated like H atoms
 - For each nitrogen atom, add one to the number of carbon and hydrogen atoms
- By calculating the IHD, we can tell from the molecular formula how many multiple bonds and rings are present in the molecule. For compounds that contain other atoms other than hydrogen:

IHD	Multiple bonds/rings present in molecule
0	Single bonds
1	Double bond/Ring structure
2	Triple bond

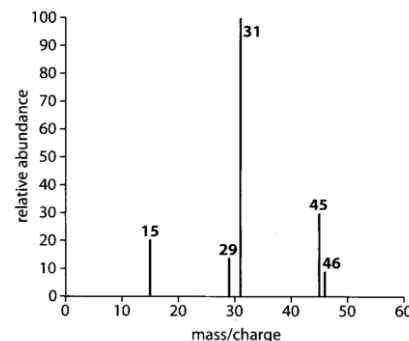
Infrared Spectroscopy

- When molecules absorb energy in the IR region of the electromagnetic spectrum, it causes the bonds between the atoms to vibrate (the bonds stretch and bend)
- The frequency of IR radiation that is absorbed is measured as the number of waves per centimeter
- The fingerprint region can be used to identify an unknown compound by comparing with the IR spectra of known compounds
- However a bond will **only interact with IR radiation if it is a polar covalent bond** (non-polar bonds do not absorb IR radiation)
- The intensity depends on the dipole moment of the bond:
 - Strongly polar bonds produce strong bands
 - Bonds with medium polarity produce medium bands
- Note IR is not generally used to determine the whole structure of an unknown molecule
- In order to analyze IR graph we need to look for:
 - Tongue: A broad, rounded peak in the region
 - Swords: These peaks are almost always the strongest peaks in the entire spectrum and are relatively narrow, giving them a somewhat "sword-like" appearance
- Once we have identified our "tongue" and our "swords" compare the wavelength value to the value in Table 26
- Note that the graph can be divided up into a functional group region, and the fingerprint region. The fingerprint region is located on the right while the functional group region is located on the left and involves the tongue



Mass Spectrometry

- While mass spectrometry is used to determine the relative atomic mass (A_r) of an element **it can also be used to determine the structure of a compound**
- Inside the mass spectrometer, some of the molecular ions break down to produce fragments
- A fragmentation pattern is produced which gives useful information about the structure of the compound
- When determining the structure of a compound don't use the mass/charge (m/z) value
- Instead take the largest value and subtract every other value from it



¹H NMR: Proton nuclear magnetic resonance spectroscopy

- NMR or nuclear magnetic resonance spectroscopy is a technique used to determine a compound's unique structure
- The position of the NMR signal is measured relative to the signal produced by TMS tetramethylsilane
- The chemical shift is measured relative to the point 0
- If there are two of the same group (two CH_3 groups), look at the groups of atoms that those groups are bonded to, if they are the same then the protons are in the same chemical environment, if not they are in different chemical environments
- First look at:
 - Number of hydrogen types
 - Peaks
 - Neighbors
 - Cause of shift