Purification

1) Physical State Separation
   - Distillation
   - Crystallization
   - Precipitation
2) Chromatography
   - Media + Adsorption

Chemical Analysis

- Qualitative Analysis
- Quantitative Analysis

Qualitative Test for Inorganic or Organic Compound

Qualitative: Determine if you have the compound of interest

Organic	Inorganic
- Contains carbon - Low mp < 200 °C, low bp - Burns frequently in air - Soluble in non-polar solvents	- No carbon - High mp & bp - "Does not burn" - Soluble in H_2O

THERE ARE MANY EXCEPTIONS!!!

E.g. Common table sugar is an organic molecule, however it dissolves in water.

Quantitative Analysis

Quantitative: How much of the compound of interest (quantity)
Amounts of atoms in compounds

$$\text{Organic compound} \xrightarrow[\Delta\ (\text{heat})]{O_2} CO_2 + H_2O + NO_2$$

$$NO_2 \xrightarrow{[H] = \text{Reduction}} N_2 + H_2O$$

MW (g/mol): CO_2 44, H_2O 18

$$\text{Compound (4.34 mg)} \longrightarrow 10.35\text{ mg} \quad 3.42\text{ mg} \quad 0\text{ mg}$$

contains C, H, O

Note: Matter cannot be created or destroyed in a chemical reaction, therefore the amount of carbon in the CO_2 is equal to the amount of carbon in the starting sample.

Percent Composition

Weight of carbon (in sample) = $\frac{12 \text{ g/mol of C}}{44 \text{ g/mol } CO_2}$ x 10.35 mg of CO_2 = 2.82 mg of C

Weight of hydrogen = $\frac{2(1 \text{ g/mol of H})}{18 \text{ g/mol of } H_2O}$ x 3.42 mg of H_2O = 0.383 mg of H

Weight of oxygen = 4.34 mg – (2.82 mg of C + 0.383 mg of H) = 1.14 mg of O

Now one can calculate percentage composition:

% Composition:

% C = $\frac{\text{Mass of carbon}}{\text{Mass of sample}}$ x 100% = $\frac{2.82 \text{ mg of C}}{4.34 \text{ mg}}$ x 100% = 65.1%

% H = $\frac{0.383 \text{ mg of H}}{4.34\text{mg}}$ = 8.83%

% O = 100% - 65.1% - 8.83% = 26.1%

The empirical (and with additional data, molecular formula) can be determined from % composition

Determining the empirical experimental formula:

Definition: Empirical formula is the ratio of atoms to each other in a molecular formula

There are three steps to calculate the empirical formula:

1) Divide each percentage (%) by the atomic weight of the element → crude ratio
2) Divide each crude ratio by the smallest crude ratio → refined ratio
3) Multiply the refined ratio by an integer value (x2, x3, x4…) → integral ratio

% Composition	Crude Ratio	Refined Ratio	Integral Ratio
65.1 % C	65.1 / 12.0 = 5.42 (% C / At Wt C)	5.42 / 1.63 = 3.34	3.34 x 3 = 10
8.83 % H	8.83 / 1.01 = 8.76	8.76 / 1.63 = 5.39	5.39 x 3 = 16
26.1 % O	26.1 / 16.0 = 1.63	1.63 / 1.63 = 1.00	1.00 x 3 = 3

From the integral ratio, the empirical formula is $C_{10}H_{16}O_3$. Using this formula an empirical weight can be calculated.

C: $10 \times 12 = 120$ g/mol
H: $16 \times 1 = 16$ g/mol
O: $3 \times 16 = 48$ g/mol

$C_{10}H_{16}O_3 = 184$ g/mol

Note: Suppose the molecular weight is given as 368 g/mol, then the molecular formula is obtained by multiplying the integral ratios by a factor of 2 and it would be $C_{20}H_{32}O_6$. The molecular weight can be independently determined via mass spectrometry.

Gas Law: (Different kinds of units for pressure and volume can be used provided the value of the gas constant is adjusted to those units)

PV = nRT P = Pressure in atm
V = Volume in L
N = Moles
T = Temperature in °K; K and °C are the same size, but 0 K = - 273 °C

$$R \text{ is a constant } \frac{0.082\ \text{L} \cdot \text{atm.}}{\text{mol} \cdot \text{K}}$$

Standard conditions for temperature and pressure (STP)
Standard pressure is 1 atmosphere, or 760 mmHg; standard temperature is 273 K
1 mol of gas occupies 22.4 L at STP

Sample Question: What volume will 3 mL of N_2 gas occupy at standard pressure and temperature (STP)?

$$\frac{P_1V_1}{P_2V_2} = \frac{nRT_1}{nRT_2} \quad \text{divide equations to give} \quad \frac{P_1V_1}{P_2V_2} = \frac{T_1}{T_2}$$

$P_1 = 760$ mmHg $\quad T_1 = 273$ °K
$P_2 = 750$ mmHg $\quad T_2 = 298$ °K $\quad V_2 = 3$ mL

Solve for V_1

$$V_1 = \frac{T_1P_2V_2}{T_2P_1} = \frac{(273\ °\text{K})(750\ \text{mmHg})(3\ \text{mL})}{(298\ °\text{K})(760\ \text{mmHg})} = 2.71\ \text{mL}$$

Question: How many moles of N_2 is 2.71 mL at STP and what is its mass?

Note: 1 mole of an ideal gas occupies 22.4 L at STP.

$$2.71 \times 10^{-3}\ \text{L} \times \frac{1\ \text{mole}}{22.4\ \text{L}} = 1.21 \times 10^{-4}\ \text{moles of } N_2$$

$$1.21 \times 10^{-4}\ \text{mol} \times 28\ \text{g/mol} = 3.4\ \text{mg of } N_2$$