Gravimetric Analysis

- Gravimetric analysis uses the mass of a sample to determine the amount of substance of interest
- Precipitation is the most common form of gravimetric analysis:

  $Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$

  - Since K_{sp} is small ($K_{sp}[AgCl] = 1.8 \times 10^{-10}$) most of the silver will be removed from solution in the form of precipitate
  - From molecular formula and mass of AgCl, amount of Ag^+ originally in solution can be calculated

Gravimetric Analysis

- Accuracy depends on several factors:

1. Value of K_{sp}—if K_{sp} is larger, more analyte will remain in solution and not be included in mass of precipitate
2. Crystal formation—crystals grow in two stages
   1. Nucleation—rapid combination of molecules to form nanoscale aggregates
   2. Particle growth—addition of more molecules to form "macroscopic" particles larger than μm scale

Gravimetric Analysis

- Potential problems with particle formation

1. Colloid formation—a colloid is a nanoscale particle (diameter < 1 μm) that remains suspended in solution
   - Colloids are recognized by "cloudy" appearance of solution—colloids are small enough to remain suspended by collisions with solvent molecules
   - Colloids are small enough to pass through filtration device and not be included in mass determination
2. Elevated temperature—most salts have higher solubility with increasing temperature

Gravimetric Analysis

- Potential problems with particle formation

3. Co-precipitation—inclusion of impurities in precipitate
   - Random occupation of sites in crystal lattice by undesired species—occurs when impurity has similar size and charge to desired analyte
   - Pockets of impurities trapped with crystal structure

Gravimetric Analysis

- Solutions to potential problems

1. Digestion—allow precipitate to form slowly in presence of hot solvent that cools gradually
   - Slow crystal formation promotes large particles
2. Washing—filter precipitate and wash with pure solvent, then re-dissolve precipitate (heating) and allow precipitate to recrystallize slowly
   - Equilibrium will favor formation of purer crystals
3. Masking agents—species that complex with impurities to keep from forming precipitate
   - CN^- complexes with Mn^{2+} in analysis of Ca^{2+}

Combustion Analysis

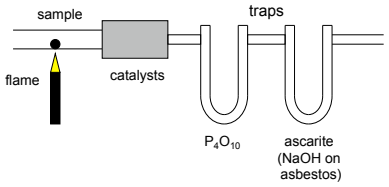
- Method to determine amount of C, H, N, and S in sample
- Sample is burned in excess oxygen and the resulting products are collected:

  $\text{sample} + O_2(xs) \rightarrow CO_2(g) + H_2O(g) + N_2(g) + SO_2(g)$

  - Catalysts are used to ensure completion reaction to the desired products
  - $C \xrightarrow{WO_3} CO_2(g)$
  - $SO_3(g) \xrightarrow{Cu} SO_2(g)$

Combustion Analysis

- Analysis methods:



P_4O_{10} absorbs H_2O generated

Ascarite absorbs CO_2 generated

Mass of CO_2 and H_2O is determined by weighing traps and taking the difference

Combustion Analysis

- Analysis methods:

Flash combustion followed by analysis using gas chromatography (GC)

- Small amount of sample is combusted in injector of GC instrument
- GC separates product mixture into individual components
- GC detector measures amount of each product

We will discuss GC in more detail later in the course (See Figure 27-5 in text for schematic diagram)