Experimental Chemistry

AP Chemistry (Barron's C14)

Table of Contents

Data Gathering
Calculations
Graphs
Determination of Physical Properties

Sample Manipulations Separation Techniques Instrumental Techniques
Experimental Reactions
Qualitative Analysis of Inorganic Ions
Chemical Hazards

Safety Principles and Equipment

Data Gathering

- Quantitative VS Qualitative
 - Quantitative \rightarrow numerical (ex concentration of acid it .345 M)
 - Qualitative → not numerical (ex when silver nitrate is added to a solution and a white precipitate)

Calculations

- 2 basic approaches
 - Dimensional analysis → converts information from one set of units to another
 - Memorized equation or law where you just plug n' chug
- Accuracy VS Precision
 - Accuracy → closeness between the measurement obtained and the true value
 - Generally it's impossible to determine the accuracy with certainty
 - Measure value using two independent methods to have more confidence in accuracy of results
 - Precision → closeness of repeated measurements to each other
 - All the values recorded are extremely similar (note that they CAN be very inaccurate though)
 - Precise ≠ Accuracy
 - Measure of **indeterminate errors** errors that arise in estimating the last, uncertain, digit of a measurement; random errors and cannot be eliminated
- **Significant figure** rules

- Significant if: digit is not zero, zero is in between two other numbers, trailing zero is in a number w/ decimal point
- Not significant if zeros are all to the left of all nonzero digits
- Exact numbers → have no uncertainty and are exactly known (ex one dozen of eggs is 12 eggs, not 12.001); considered to have infinite sig figs when doing math
- Math rules
 - Multiplication and division number with fewest significant figures determines number of sig figs in answer
 - Addition and subtraction number w/ fewest decimal places determines the number of decimal places in answer

Uncertainty

- Absolute uncertainty uncertainty of the last digit of a measurement (45.47 mL last digit is uncertain) (for addition/subtraction)
- Relative uncertainty absolute uncertainty divided by the number itself (for multiplication/division)

Rounding

- Number of digits to be kept determined with sig fig rules
- If number after kept digits is left than 5 → round down
- o If number are kept digits is 5 or more → round up
- Significant figures in atomic and molar masses
 - Make sure to first determine the number of sig figs the answer must have

Graphs

- Graph → used to illustrate the relationship between two variables w/ two aces (x-axis and y-axis)
- X-axis is normally the independent variable while y-axis is normally the dependent variable
- Make sure to remember to label the axes, number the axes, plot the data points, and (not always) draw the line of best fit

Determination of Physical Properties

Scale reading

- Parallax errors fluid level or meter pointer is never directly in contact with scale, incorrect reading results leads to parallax errors
- Meniscus surface tension of a liquid in any container causes liquid to have curved surface

ALWAYS MEASURE BOTTOM OF MENISCUS

Determination of mass by weighting

Weight = gravitational constant * mass

- Proper use of balance requires that balance is calibrated sample is always weighed in an appropriate container (never directly on the balance pan)
- Tare mass mass of the empty container used to hold whatever you are measuring

• Liquid volume measurement

- Pipettes or burettes are used for more accurate liquid measurements + graduated cylinders and graduated beakers are used for larger, less precise measurements
- Labels
 - **TD** = to deliver
 - **TC** = to contain (ex the volumetric flask)
- Burets
 - Long, graduated tubes that hold between 10 to 50 mL of solution
 - Flow of liquid is controlled by a valve called a **stopcock**
 - Volume of soln delivered = volume at end volume at start

• Temperature measurement

- Thermometers are used to measure temperature
- Calibration of thermometer checked by immersing in ice water (0 degrees)
 and boiling water (100 degrees)

• Density determination

- Density = mass / volume
- Density of solid or liquid = grams of material / cm³ of material
- Density of gases = grams of gas / liters of gas
- Pycnometer used to determine density of liquid small flask with volume of approx. 5-25 mL

• Determination of specific heat

- Specific heat of solid (usually metal0 determined by heating a known mass of substance to predetermined temperature and then submerging it into a known quantity of water in an insulated container
- Q = mass * specific heat * temperature

Sample Manipulations

- Cool using an ice bath of crushed ice and water
- **Volumetric flask** calibrated with an etched line on its neck to hold a specified volume at given temperature
- ALWAYS ADD ACID TO WATER TO PREVENT BAD TINGZ FROM HAPPENING

Dilution

Initial concentration * initial volume = final concentration* final volume

• **Pneumatic trough** - when gas is generate in chemical reaction, it can be collected in a pneumatic trough

Separation Techniques

• Precipitation

- Solids can be precipitated using chemical reactions that produce solid from reactants that dissolve in water
- Once precipitation is complete, mixture is heated to coagulate small crystals into large crystals for easy filtration

Filtration

- Used to separate solid particles from a liquid
- Filter paper is folded into cone and inserted into a funnel, where a few drops of water are placed on the filter paper to hold in place and then solution to be filtered is added
- Precipitates are washed with dilute electrolyte solutions to remove contaminants

Centrifugation

- For small amounts of precipitate
- Spins samples at high speed, forcing solids to compact at bottom of test tube
- **Supernatant** the clear liquid that remains after centrifugation

Distillation

- Separation technique where substance w/ high vapor pressure (low boiling point) is separated from other substances with lower vapor pressures (higher boiling points)
- Performed by boiling a solution and passing a solution and passing the vapor formed through a condenser to recover the vaporized liquid

Instrumental Techniques

• pH determination

- pH meter used when precise pH value is needed → has glass and reference electrode, standardized with a standard buffer, electrodes rinsed to remove any buffer and immersed in sample
- o **pH paper** serves to estimate the pH of solution
 - **Litmus paper** used to tell whether something is acid or basic
 - Pink is acidic and blue is basic

Spectroscopy

 Spectrophotometer - colored solutions absorb visible light, which can be measured which a spectrophotometer

- **Absorbance** of a sample is quantity determined in a photospectrometer
- o spectrum is the graph of light absorbed by a light absorbed by a sample
- Concentration of solute determined at the wavelength of the largest peak in the spectrum
- Beer's law says that absorbance = absorptivity * optical path length *
 concentration
 - \blacksquare A = a * b * c
 - Absorptivity is a constant value characteristic of compound and wavelength at which absorbance is measured
 - Optical path length is the thickness of the sample
 - Slope of graph of absorbance as function of concentration is a * b

Experimental Reactions

• Gas synthesis

- Some gases synthesized and collected include O2, H2, CO2, NO, H2S, NH3
- Gases that don't react or dissolve in water can be measured by calculated the displacement in a pneumatic trough

• Insoluble salt synthesis

- Often precipitated in double-replacement reactions or in reactions of gases with soluble substances
- Insoluble carbonates and sulfites are formed by bubbling CO2 or SO2 through solutions containing aqueous metal ions

• Soluble salt preparation

- Soluble salts isolated from aqueous solution bee removing water via evaporation or applying heat
- To obtain a pure sample of salt, the solution must contain only the ions of that salt
- Can be prepared via neutralization reactions as long as neither an excess of acid nor an excess of base is used in reaction

Qualitative Analysis of Inorganic Ions

• When separation is by filtration, the liquid is called the **filtrate**; when the precipitate is separated by centrifugation, liquid phase is called **supernatant**

Chemical Hazards

- COMMON SENSE BE CAREFUL WHILST DOING LABS
- Keep organic compounds and etheres away from sparks and flames, be careful of ethers reacting over time to form explosive peroxides, strong oxidizers that cause skin injury, alkali metals that react explosively in water, etc.