Quick Guide to Experimental Procedures

Gravimetric Analysis:

Weighing the sample to be analyzed.	**Dissolving this sample in water.**	**Adding a suitable chemical to form a precipitate.**	**Filtering to collect the precipitate, Rinsing of precipitate to remove soluble ions.**	**Repeated heating and weighing until a constant mass of precipitate is obtained.**

Common Mistakes:

- **Precipitate is not dry when you take the final mass.**
  - Results in the appearance of more precipitate than was actually produced because some mass is water.
  - Percent yield would be higher than it should be.

Common Applications:

- Mixtures of solids—determining the amount of a particular ion in a solution

Important to Remember:

- All sodium, nitrate, ammonium, and potassium compounds are soluble. Net ionic equations would not include these ions.

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Making a solution:

The AP Readers would prefer that you use a pipet at this point to make sure you hit the line perfectly.

(a) An amount of solute is weighed out on an analytical balance and then **(b) A portion of the solvent is added to the volumetric flask.** **(c) The mixture is swirled until *all* of the solute is dissolved.** **(d) Additional solvent is added up to the mark on the volumetric flask.**

Common Mistakes:

- **Solid gets stuck in the neck of the flask**
  - Use a beaker to dissolve solute in some solvent, then transfer to volumetric flask.
- **Overfilling the volumetric flask**
  - Results in a dilute solution
- **Not using distilled water.**
  - Other ions could affect the experiment for which the solution is used
- **Not using a volumetric flask (beaker or Erlenmeyer instead)**
  - Loss of precision in concentration of prepared solution

Common Applications:

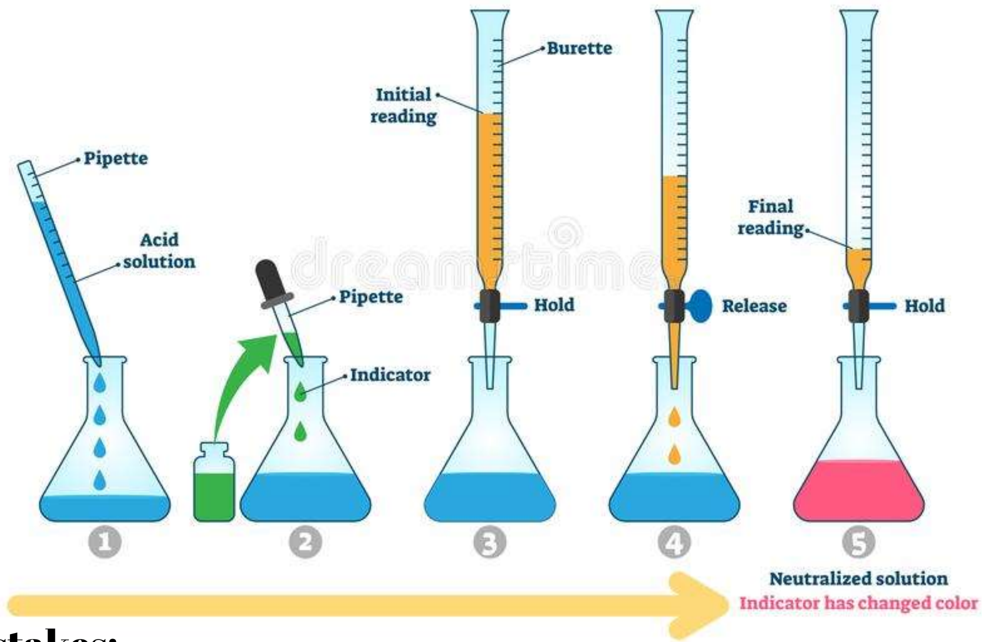
- Making solutions to dissolve substances for analysis, particularly in titrations.

Important to Remember:

- Molarity = moles solute/L of solution

Quick Guide to Experimental Procedures

Titration:



Common Mistakes:

- **Overshooting the titration (too dark of a color at the end)**
  - Results in the concentration of the unknown solution in the flask appearing to be higher than it actually is, since more titrant must be added.
- **Not using indicator.**
  - No perceivable endpoint.
- **Using incorrect indicator.**
  - pH at the equivalence point should be approximately equal to the pKa of the indicator.
- **Cleaning and preparing the buret incorrectly.**
  - Rinse buret with distilled water, add a small amount of titrant to buret, swirl, and let it out through the stem.
  - Consequence of improper cleaning will be a titrant that is more dilute, which will result in an analyte that appears to be more concentrated than it is
- **Reading buret incorrectly**
  - It should be read from the bottom of the meniscus. If on the line, add a 0, if inbetween, estimate the final digit

Common Applications:

- Solving for the concentration of an unknown substance (analyte).
- Acid/Base, Redox

Important to Remember:

- Molarity = moles solute/L of solution
- **Analyte:** substance in flask
- **Titrant:** substance in buret
- **Standard solution:** solution of known concentration, usually goes into the buret.
- $\mathbf{M_1V_1 = M_2V_2}$ is helpful for solving for the concentration of the analyte solution at the equivalence point (**if the acid is monoprotic**)
- **For polyprotic acids** use stoichiometry to determine concentration of unknown
- **Endpoint:** point in titration where flask solution changes color
- **Equivalence point:** point in the titration where the moles of acid are equal to the moles of base

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Analyzing Concentration of Solutions Using Beer's Law):

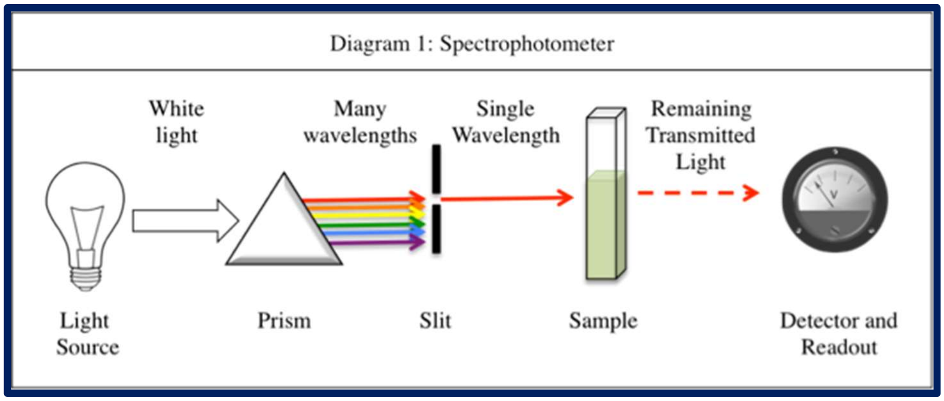


Diagram 1: Spectrophotometer

Step 1: Pick the wavelength for the solution where absorbance is highest (for solute). Complementary colors are usually best.

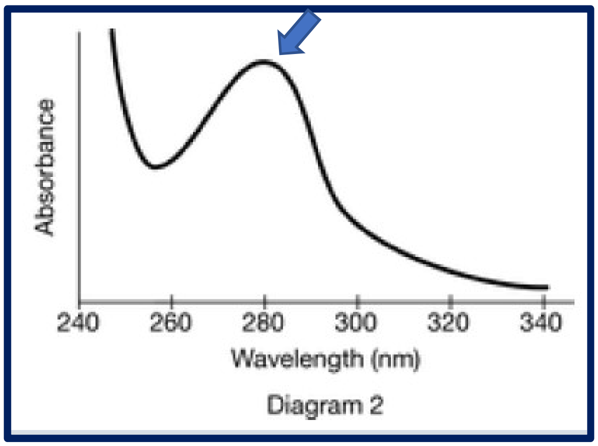
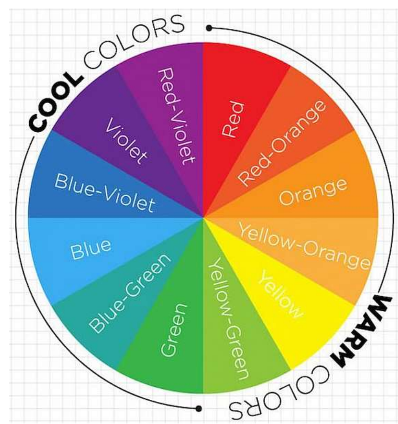
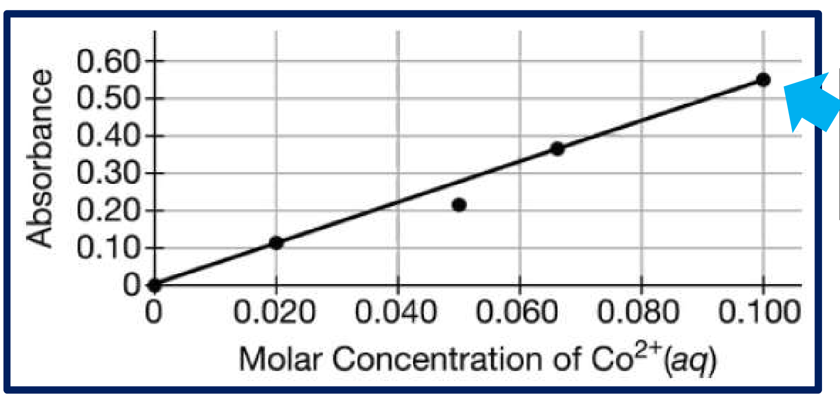


Diagram 2



Step 2: Measure absorbance for different concentrations at that wavelength. Graph results.



Concentration and Absorbance are directly proportional.

A = εbc

Absorbance = (molar absorptivity)(cuvette pathway length)(concentration)

Quick Guide to Experimental Procedures

Common Mistakes:

- **Absorbance is lower than it should be (point falls below the line)**
    - Cuvette was cleaned with distilled water and then immediately filled with solution, creating a more dilute solution
    - Too little solute in the prepared solution
- **Absorbance is higher than it should be (point falls above the line)**
    - Cuvette is dirty with fingerprints/dust, etc.
    - Too much solute in the prepared solution
    - Contamination with a more concentrated solution
    - Used a cuvette with a longer path for one data point
    - Used frosted/ridged side of cuvette instead of the clear side
- **Did not use the correct wavelength of maximum absorbance for the solute.**
    - Absorbances could be too low especially for dilute solutions
- **Overfilled the cuvette**
    - Should not have an impact on data
- **Picked a wavelength where it is high absorbance for the solvent**
    - Won't be able to distinguish absorbance due to solvent vs. solute

Common Applications:

- Determining the concentration of a solution of unknown concentration using solutions of known concentration
- Kinetics reactions (like bleach + blue food dye)

Important to Remember:

- Before using, you need to calibrate the spectrophotometer with a blank of just solvent (in order to account for any absorbance due to solvent and cuvette itself)
- Molarity = moles solute/L of solution
- **Absorbance** is the amount of light the solution absorbs at a specific wavelength
- **Molar absorptivity** (1/M*cm) describes how intensely a sample absorbs light at a specific wavelength (constant unique to the substance at a specific wavelength)
- **Path length** of sample is the length of the cuvette where the light will travel (cm)
- Concentration is molarity

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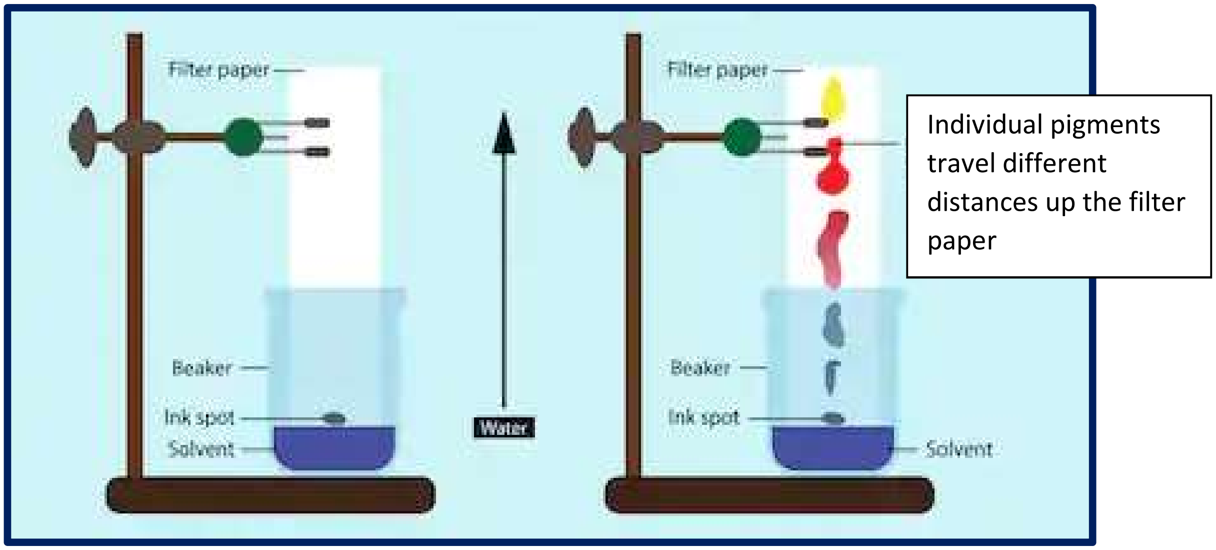
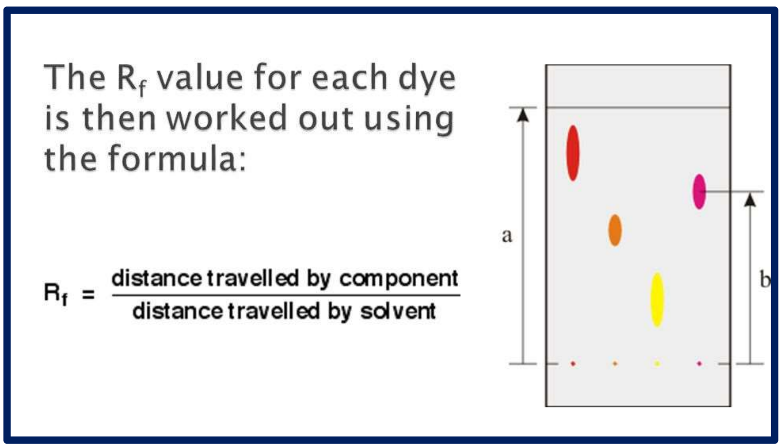
Chromatography





Common Mistakes:

- **Solvent reaches the top of the paper strip.**
    - Rf values cannot be calculated as we do not know how far the solvent would have traveled had there been more paper.
- **No major difference in polarity between paper and solvent**
    - Substances cannot be adequately separated
- **No major differences in polarity of components of mixture**
    - Substances cannot be adequately separated

Common Applications:

- Determining the components of a mixture

Important to Remember:

- Paper is usually relatively nonpolar in comparison to the solvent.
- The substance that travels further up the paper is more attracted to the solvent.
- The substance that travels the least is most attracted to the paper.
- If multiple trials are run, compare Rf values, not relative heights.
- **Polar substances** tend to lack symmetry, have polar bonds, and have lone pairs on the central atom. They are most soluble in other polar substances.
- **Nonpolar substances** tend to be symmetrical, have identical bonds, and have no lone pairs on the central atom. They are most soluble in other nonpolar substances.

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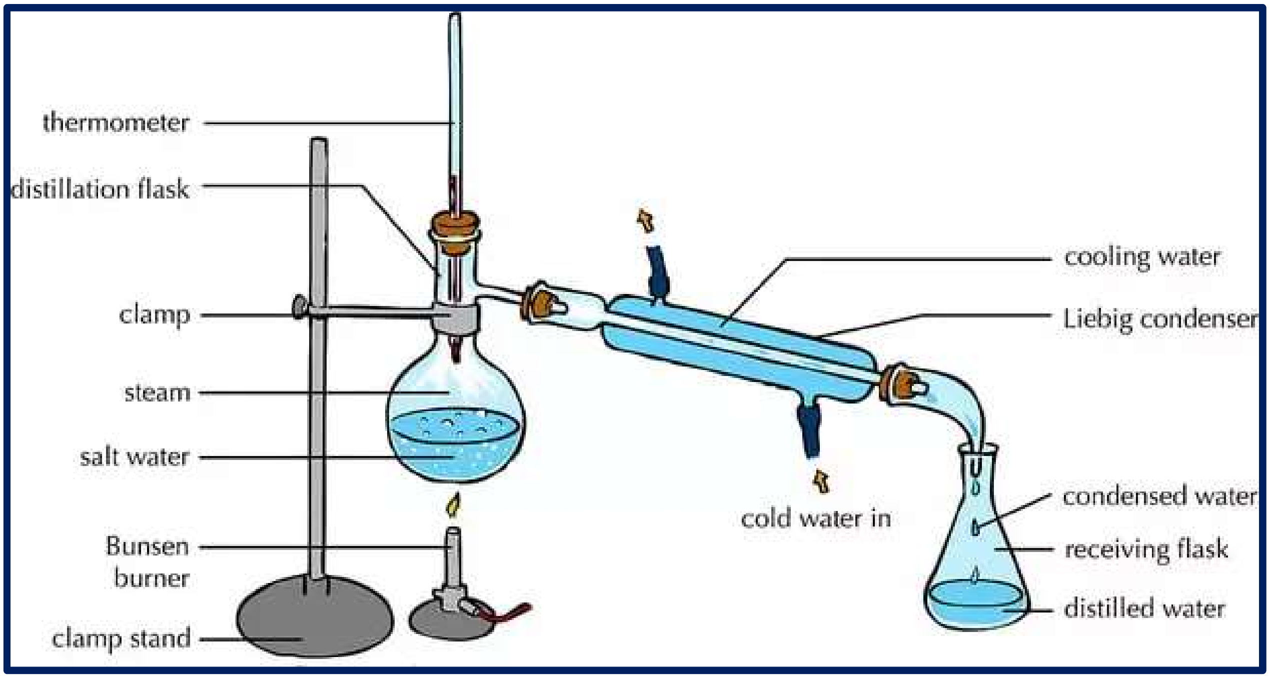
Fractional Distillation



Common Applications:

- Separating components in a solution/mixture based on differences in boiling point

Important to Remember:

- Substance collected in the flask at the end is the **distillate** (substance with lower boiling point)
- The substance with the **lower boiling point** has a **greater** vapor pressure and **weaker** intermolecular forces
- The substance with the **higher boiling point** has a **lower** vapor pressure and **stronger** intermolecular forces
- The temperature of the solution will remain constant while a component is boiling off.
- Thermometer should not be touching the bottom of the flask, or the solution will appear hotter than it actually is.

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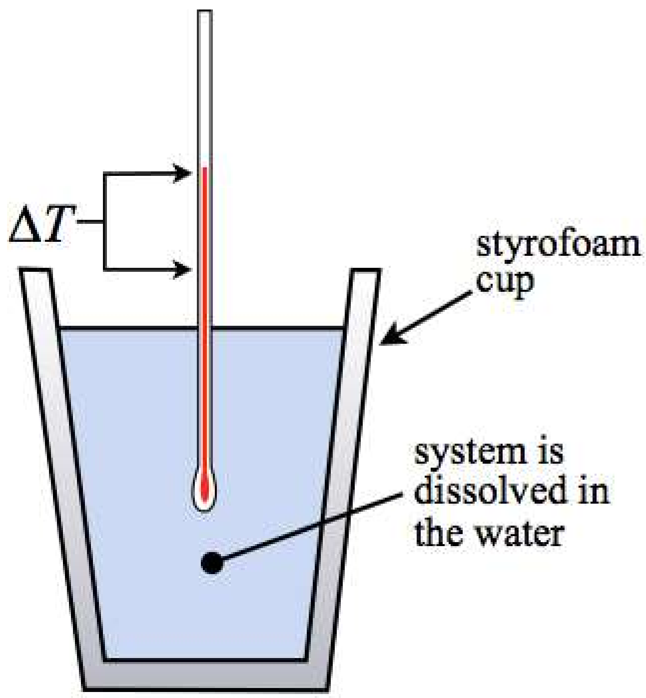
Coffee Cup Calorimetry



Common Mistakes:

- The final temperature is the highest (for exothermic) or lowest (for endothermic) temperature recorded during the reaction/process
- Not stirring enough (hotter/colder in some parts of solution)
- Endothermic reaction: temperature doesn't change enough
  - Heat was absorbed by reaction from calorimeter/surroundings
  - Lid not sealed tightly on calorimeter
- Exothermic reaction: temperature doesn't change enough
  - Heat absorbed by calorimeter or lost to surroundings
  - Lid not sealed tightly on calorimeter

Applications:

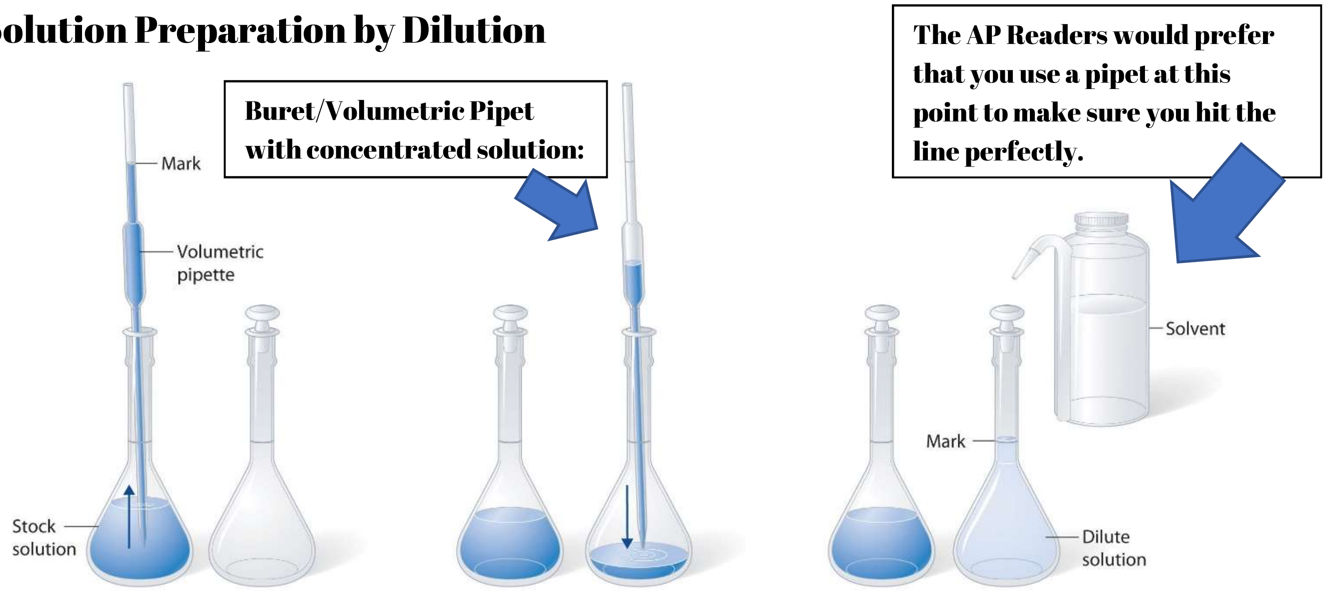
- Solving for the specific heat of a metal or the heat of reaction

Important to Remember:

- **Endothermic** processes have a **decrease** in temperature.
- **Exothermic** processes have an **increase** in temperature.
- The water is not part of the system. It is part of the surroundings.
- $q = mC\Delta T$
  - q = heat in Joules or calories
  - m = mass of entire solution (reactants + water) OR object, grams or kilograms
  - C = specific heat capacity, J/g°C (or a variation of the above)
  - $\Delta T = T_{final} - T_{initial}$
  - To calculate heat of solution: q/moles of salt
  - To calculate heat of reaction: $\frac{q}{mol\ reactant\ used} = \frac{\Delta Hrxn}{coefficient\ from\ equation}$

Quick Guide to Experimental Procedures

Solution Preparation by Dilution



(a) A volume (V_s) containing the desired moles of solute (M_s) is measured from a stock solution of known concentration.

(b) The measured volume of stock solution is transferred to a second volumetric flask.

(c) The measured volume in the second flask is then diluted with solvent up to the volumetric mark [$(V_s)(M_s) = (V_d)(M_d)$].

Common Mistakes:

- **Not adding the acid into the water (adding in reverse order)**
  - Solution can bubble up, steam can result from heat released, splattering could occur.
- **Overfilling the volumetric flask**
  - Results in a dilute solution
- **Not using distilled water.**
  - Other ions could affect the experiment for which the solution is used
- **Not using a volumetric flask (beaker or Erlenmeyer instead)**
  - Loss of precision in concentration of prepared solution

Common Applications:

- Making solutions to dissolve substances for analysis, particularly in titrations.

Important to Remember:

- Molarity = moles solute/L of solution

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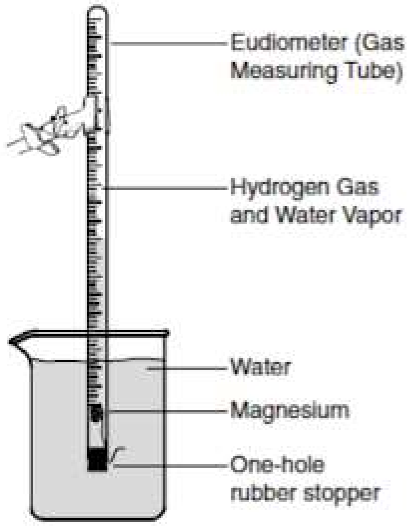
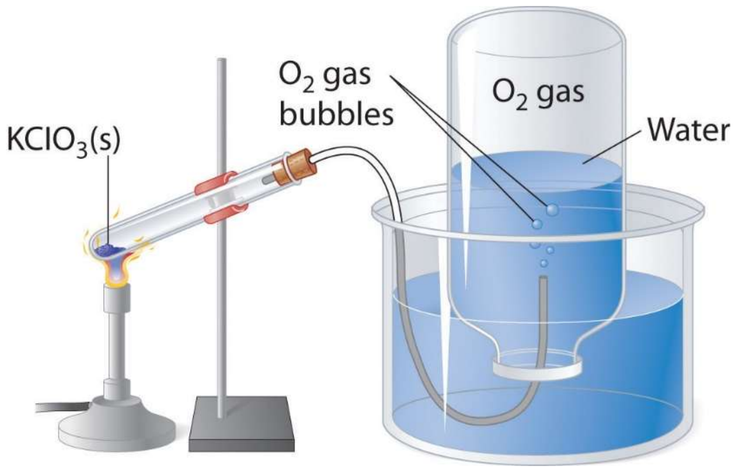
Gas Collection Over Water





Common Applications:

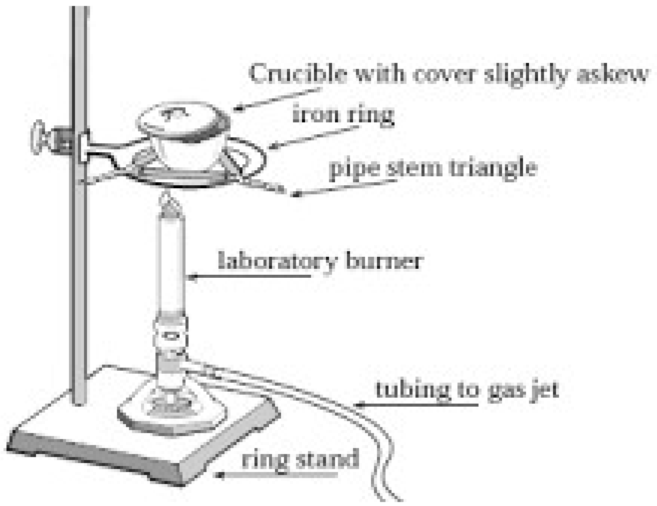
- Collecting gases that form in reactions like
  $Mg + 2HCl \rightarrow MgCl_2 + H_{2\ (g)}$
  $2KClO_3 \rightarrow 2KCl + 3O_{2\ (g)}$

Important to Remember:

- **Take the temperature of the bath to get the temperature of the gas.**
- **Use room temperature water for the bath.**
  - Gas solubility is minimized if you do not use cold water.
- **The volume of the gas must be read where the volume inside the eudiometer is at the same level as the water outside the bath.**
  - Allows the pressure inside to be equal to the atmospheric pressure
- **Pressure of atmosphere = Pressure of gas + Pressure of water vapor**

Quick Guide to Experimental Procedures

Percent Composition/Formula of a Hydrate



1. Take mass of hydrate.
2. Heat until all water has been driven off.
3. Cool, then weigh.
4. Heat again for a couple more minutes.
5. Cool, then weigh.
6. If constant mass has been reached, experiment is complete.

Common Mistakes:

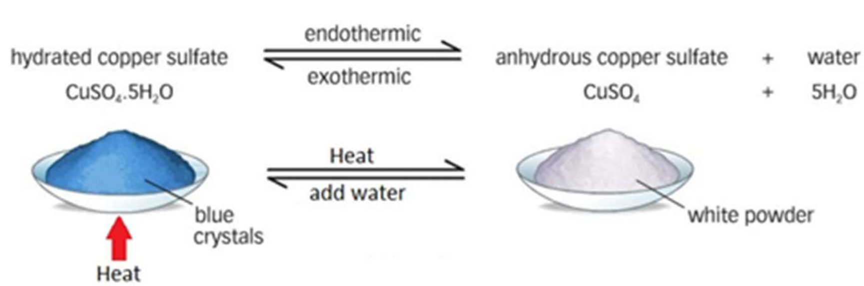
- **Not heating the hydrate enough**
    - Ratio of anhydrous salt: water will not be accurate, as water will remain in the sample
    - Appears fewer moles of water and more moles of salt will be in the hydrate
- **Overheating the hydrate**
    - Anhydrous salt could decompose in the heat
    - It will appear as though the salt is composed of more water than it is
- **Salt sticks to spatula or is spilled in the process of the lab**
    - It will appear as if there is more water in the sample than there actually is; more moles of water will appear to be in sample than there actually are
- **Crucible is weighed while still warm**
    - Inaccurate mass will be obtained

Common Applications:

- Empirical formula of hydrates, percent composition of hydrates

Important to Remember:

- Hydrated salt: before heating
- Anhydrous salt: after heating
- Moles of anhydrous salt: moles of water = ratio for hydrate



Quick Guide to Experimental Procedures

Citations for Images:

Gravimetric Analysis: https://i.ytimg.com/vi/Zi1Yh6dr03w/maxresdefault.jpg (Pittwater House School Science Department, April 2016)

Making a Solution: https://wou.edu/chemistry/courses/online-chemistry-textbooks/ch150-preparatory-chemistry/chapter-7-solutions/

Titration: https://www.dreamstime.com/illustration/chemical-setup-test.html

Spectrophotometer: https://www.varsitytutors.com/act_science-help/how-to-find-data-representation-in-chemistry?page=7

Color Wheel: https://decoart.com/blog/article/318/color_theory_basics_the_color_wheel

Chromatography: https://www.shutterstock.com/search/chromatography

Fractional Distillation: https://pediaa.com/difference-between-fractional-distillation-and-simple-distillation/

Coffee Cup Calorimetry: **https://tinyurl.com/r758672**

Making a Solution by Dilution: https://wou.edu/chemistry/courses/online-chemistry-textbooks/ch150-preparatory-chemistry/chapter-7-solutions/

Gas Collection: https://sites.google.com/a/moreaucatholic.org/ap-chemistry-labs-2011-12/stuff-of-interest/determiningthemolarvolumeofagas

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Hydrate Diagram: https://www.tutormyself.com/edexcel-igcse-2017chem-318/