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.

Quick Guide to Experimental Procedures

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:

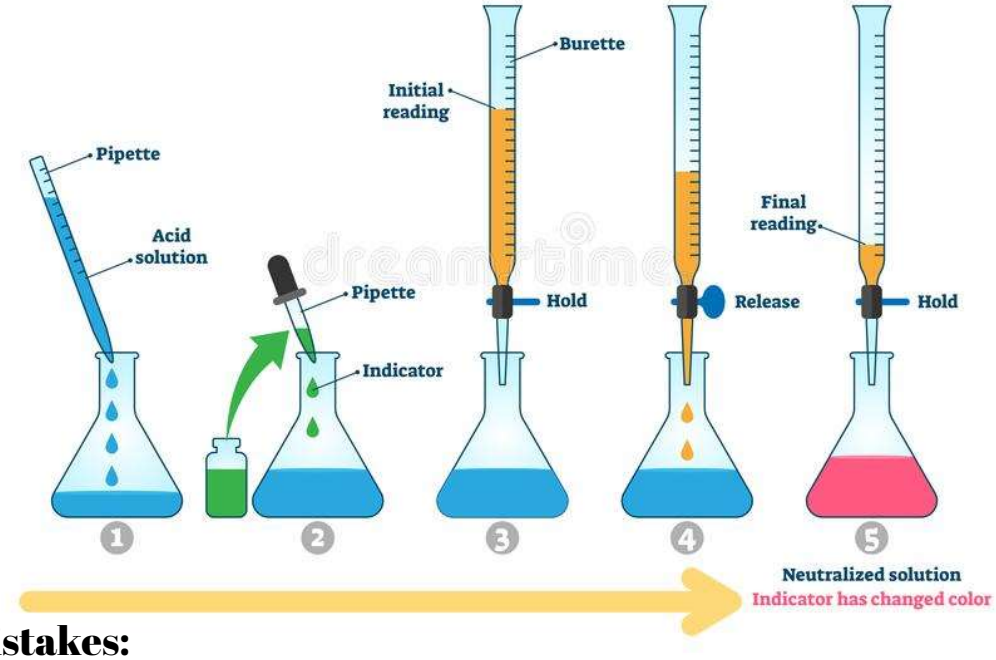
- Making solutions to dissolve substances for analysis, particularly in titrations.

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:

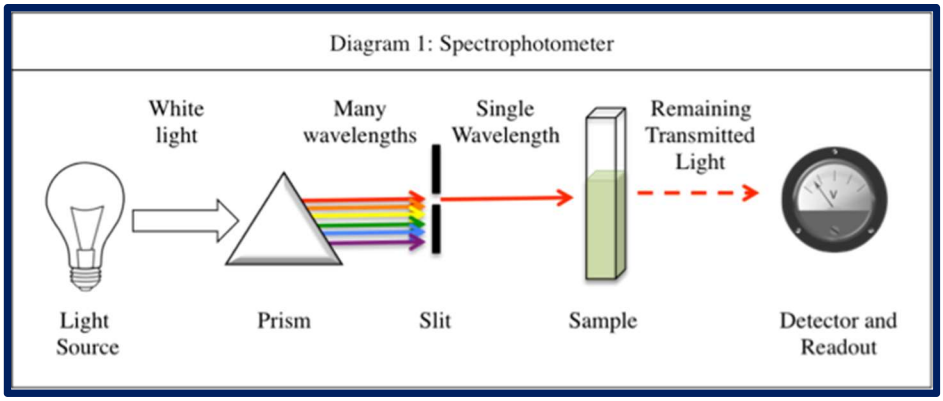
- Solving for the concentration of an unknown substance (analyte).
- Acid/Base, Redox

Important to Remember:

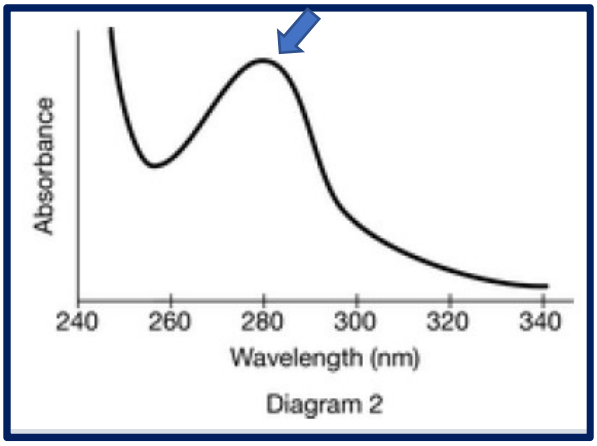
- Molarity = moles solute/L of solution
- **Analyte:** substance in flask
- **Titrant:** substance in buret
- **Standard solution:** solution of known concentration, usually goes into the buret.
- $\mathbf{M_1V_1 = M_2V_2}$ is helpful for solving for the concentration of the analyte solution at the equivalence 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

Quick Guide to Experimental Procedures

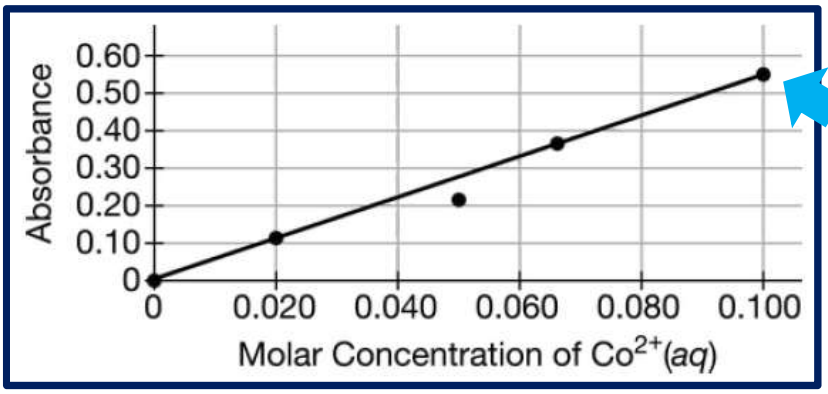
Analyzing Concentration of Solutions Using Beer's Law):



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



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



Concentration and Absorbance are directly proportional.

$A = \varepsilon bc$

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:

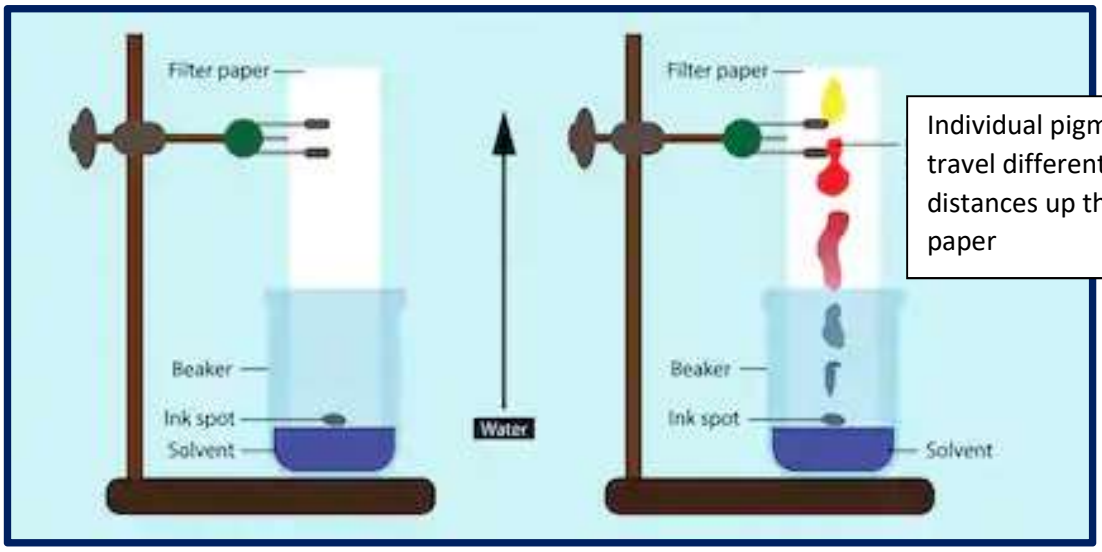
- Determining the concentration of a solution of unknown concentration using solutions of known concentration
- Kinetics reactions (like bleach + blue food dye)

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

Quick Guide to Experimental Procedures

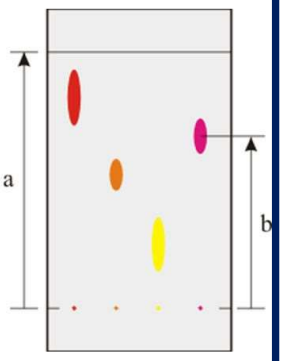
Chromatography



Individual pigments travel different distances up the filter paper

The R_f value for each dye is then worked out using the formula:

$$R_f = \frac{\text{distance travelled by component}}{\text{distance travelled by solvent}}$$



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:

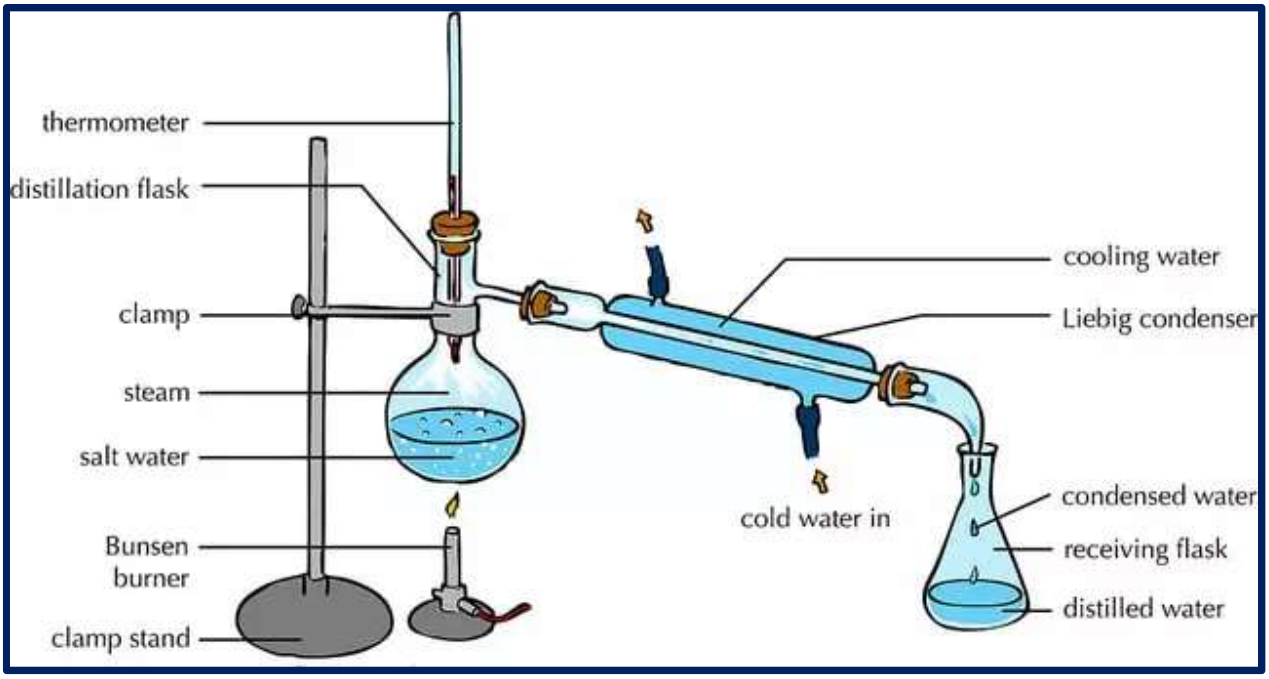
- Determining the components of a mixture

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.

Quick Guide to Experimental Procedures

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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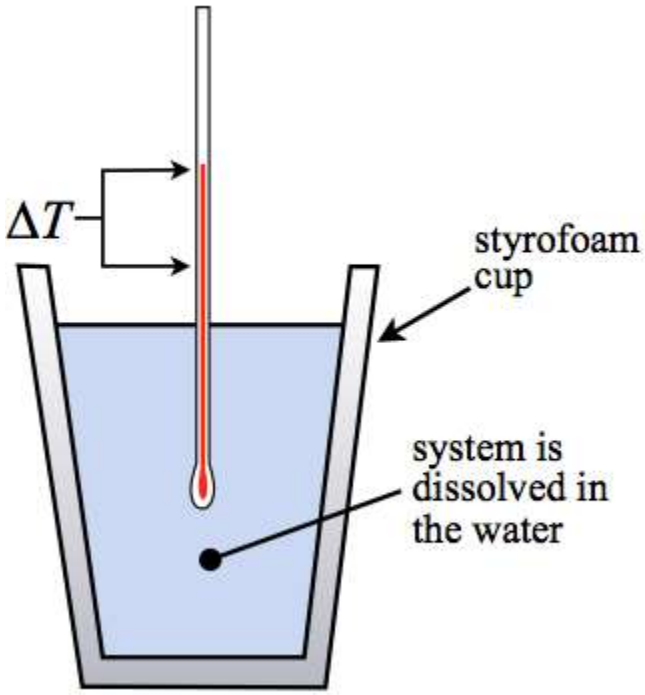
Coffee Cup Calorimetry



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:

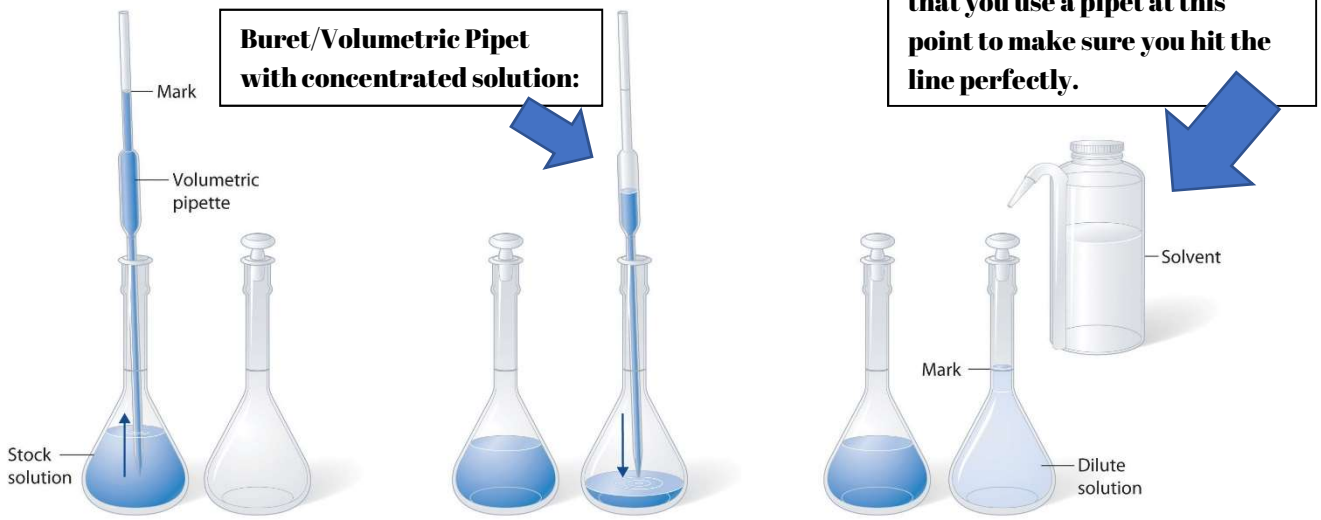
- **Endothermic** processes have a **decrease** in temperature.
- **Exothermic** processes have an **increase** in temperature.
- The water is not part of the system. It is part of the surroundings.
- $q = mC\Delta T$
  - q = heat in Joules or calories
  - m = mass of entire solution (reactants + water) 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

Buret/Volumetric Pipet with concentrated solution:

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



(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:

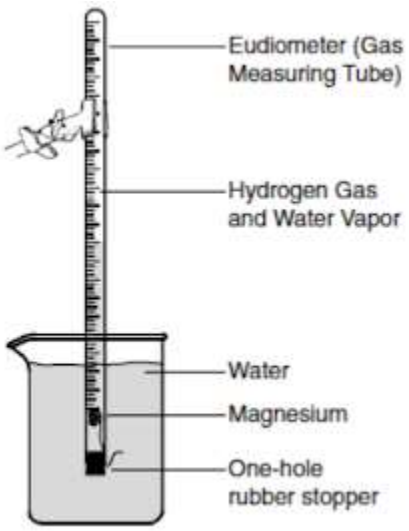
- Making solutions to dissolve substances for analysis, particularly in titrations.

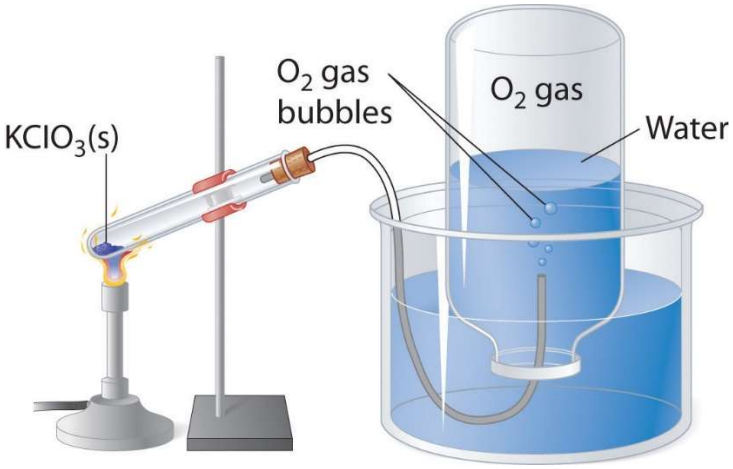
Important to Remember:

- Molarity = moles solute/L of solution

Quick Guide to Experimental Procedures

Gas Collection Over Water





Common Applications:

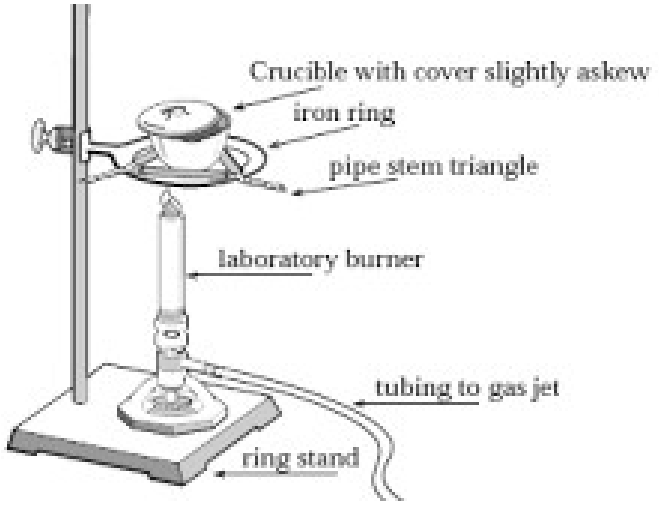
- Collecting gases that form in reactions like
  $Mg + 2HCl \rightarrow MgCl_2 + H_{2\,(g)}$
  $2KClO_3 \rightarrow 2KCl + 3O_{2\,(g)}$

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:

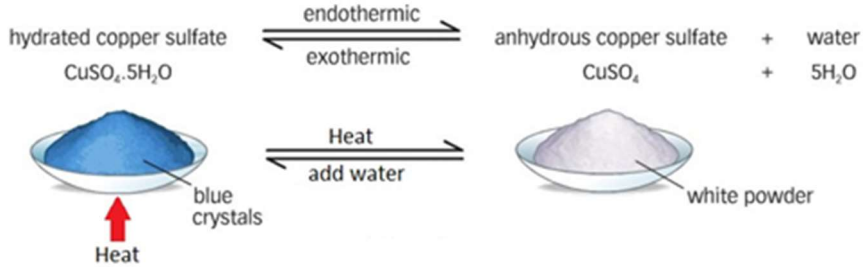
- **Not heating the hydrate enough**
  - Ratio of anhydrous salt: water will not be accurate, as water will remain in the sample
  - Appears fewer moles of water and more moles of salt will be in the hydrate
- **Overheating the hydrate**
  - Anhydrous salt could decompose in the heat
  - It will appear 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

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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/

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Crucible Set Up: https://whs.rocklinusd.org/documents/Science/Epsom_salt_lab.pdf

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