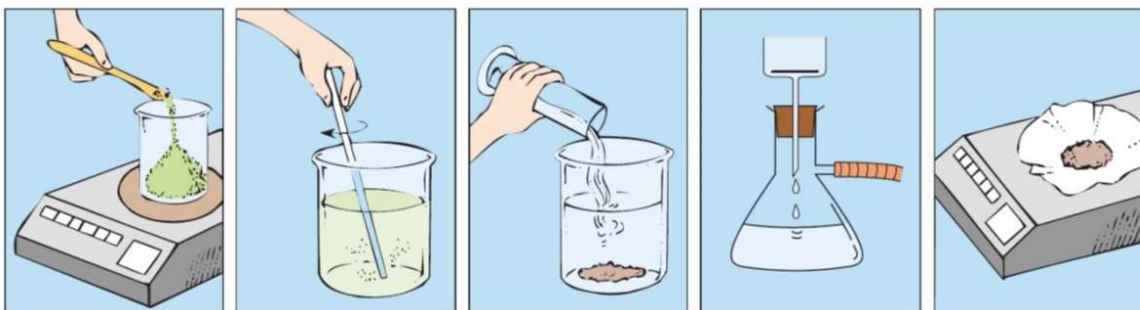


# Quick Guide to Experimental Procedures

## Gravimetric Analysis:



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

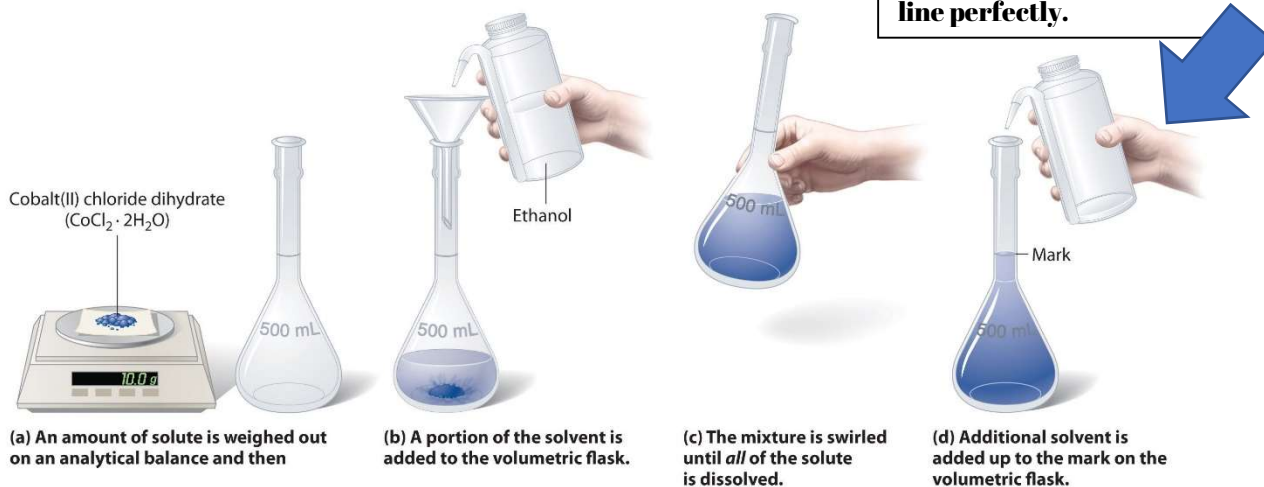
## Important to Remember:

- 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.



## 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:

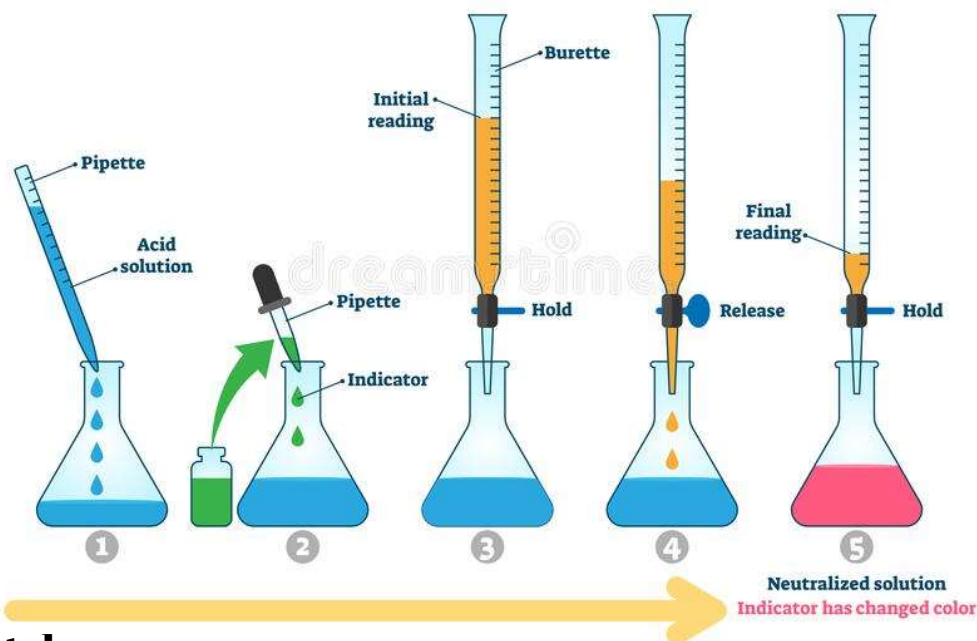
- Making solutions to dissolve substances for analysis, particularly in titrations.

## 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:

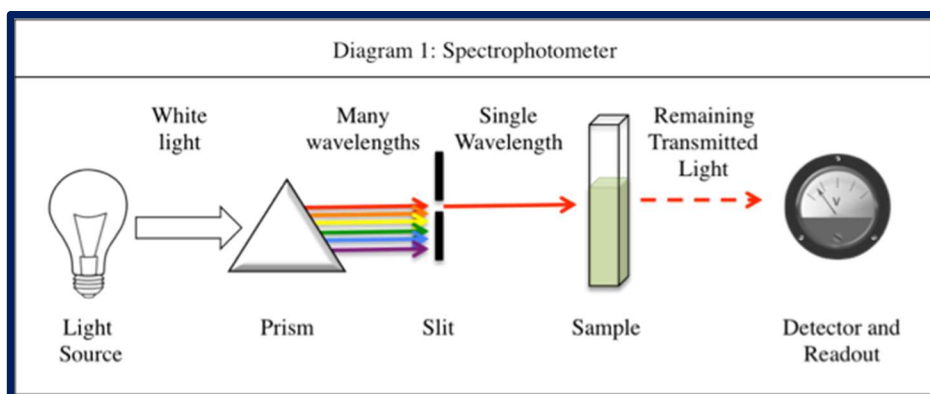
- Solving for the concentration of an unknown substance (analyte).
- Acid/Base, Redox

## Important to Remember:

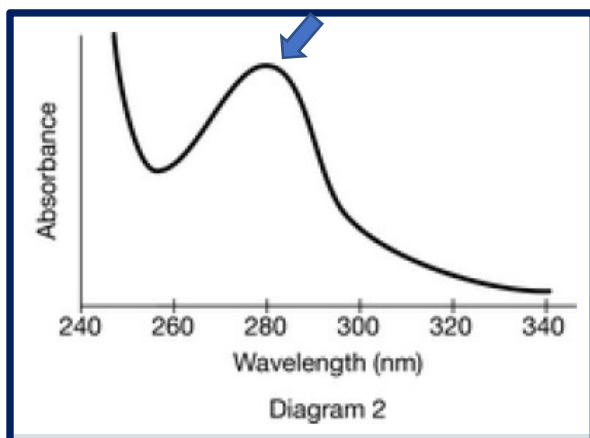
- Molarity = moles solute/L of solution
- **Analyte:** substance in flask
- **Titrant:** substance in buret
- **Standard solution:** solution of known concentration, usually goes into the buret.
- $M_1V_1 = M_2V_2$  is helpful for solving for the concentration of the analyte solution at the equivalence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

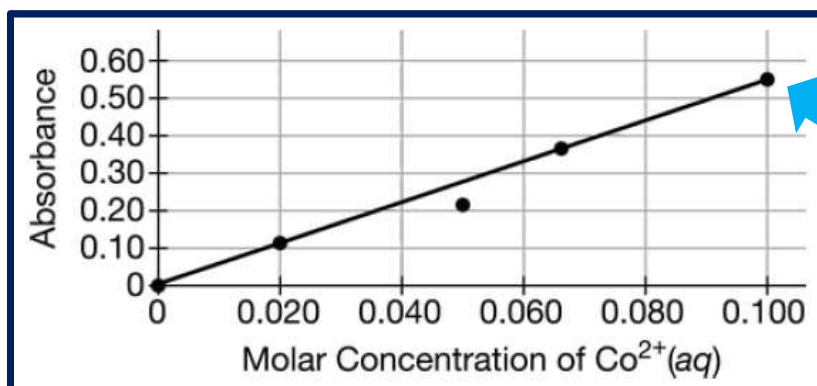
## Analyzing Concentration of Solutions Using Beer's Law):



**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.**

$$A = \epsilon bc$$

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:

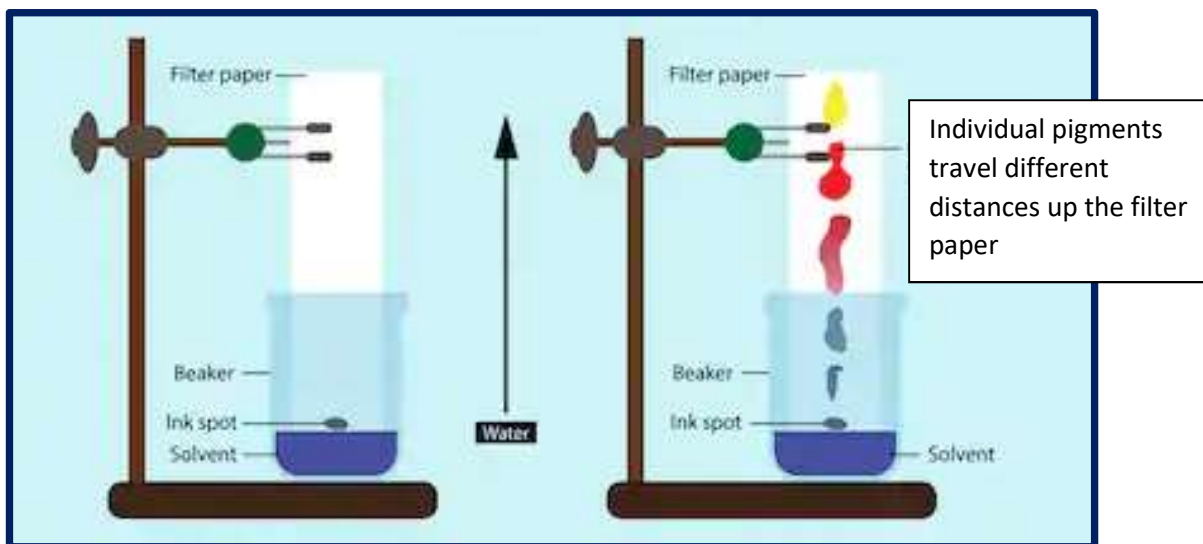
- Determining the concentration of a solution of unknown concentration using solutions of known concentration
- Kinetics reactions (like bleach + blue food dye)

## 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** ( $1/M \cdot \text{cm}$ ) 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

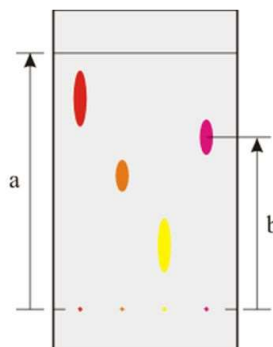
# Quick Guide to Experimental Procedures

## Chromatography



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.**
  - $R_f$  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:

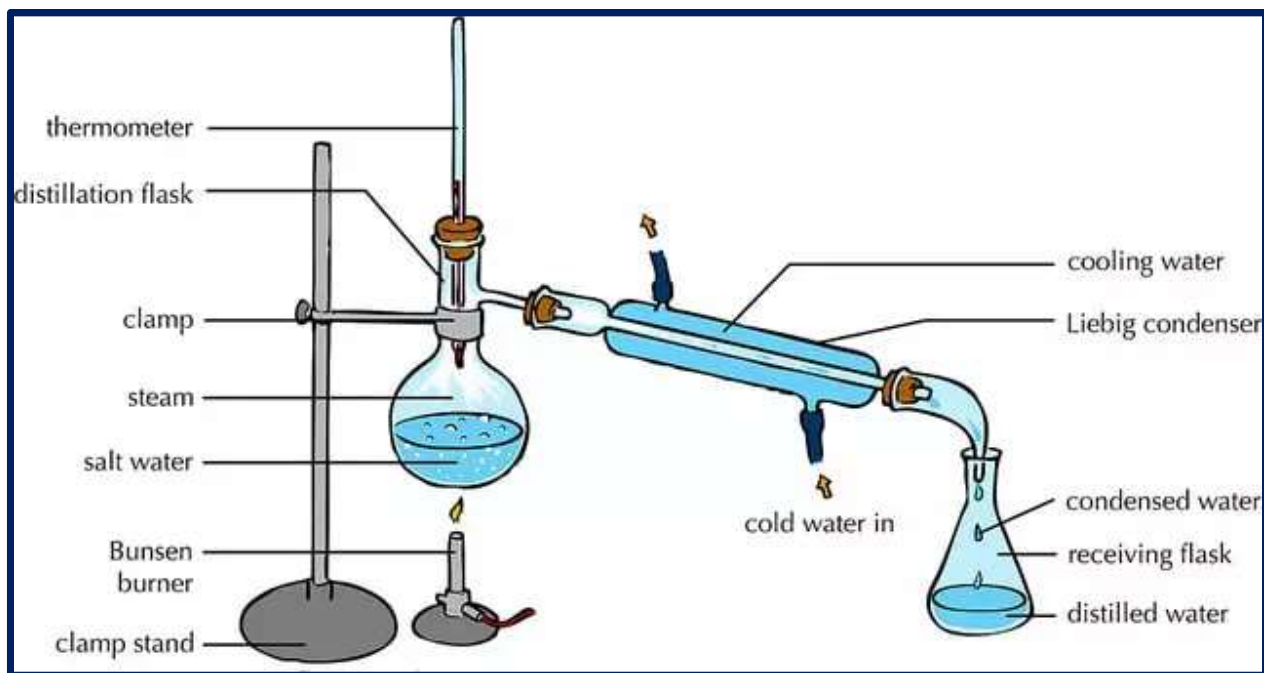
- Determining the components of a mixture

### 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  $R_f$  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:

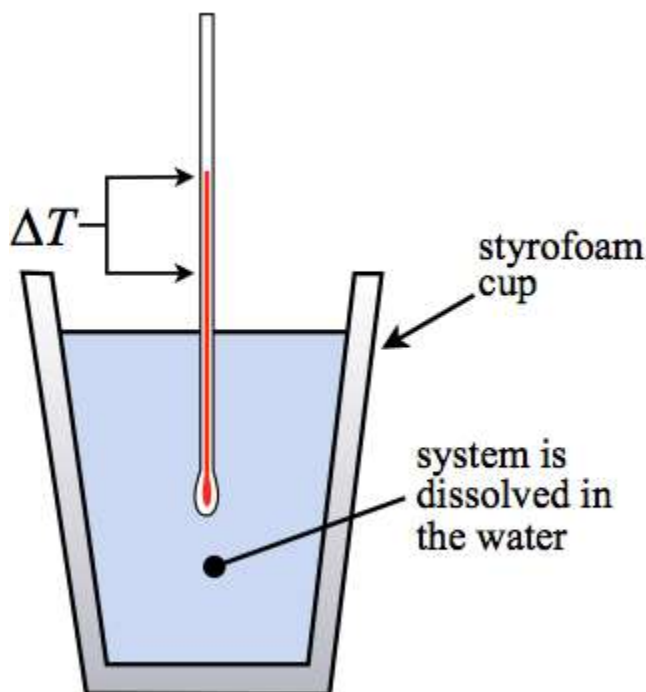
- Separating components in a solution/mixture based on differences in boiling point

### 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.

# Quick Guide to Experimental Procedures

## 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,  $J/g^{\circ}C$  (or a variation of the above)
  - $\Delta T = T_{\text{final}} - T_{\text{initial}}$
  - To calculate heat of solution:  $q/\text{moles of salt}$
  - To calculate heat of reaction:  $\frac{q}{\text{mol reactant used}} = \frac{\Delta H_{\text{rxn}}}{\text{coefficient from equation}}$



# Quick Guide to Experimental Procedures

## Solution Preparation by Dilution

**Buret/Volumetric Pipet with concentrated solution:**

(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 [ $(V_s)(M_s) = (V_d)(M_d)$ ].

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

## 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:

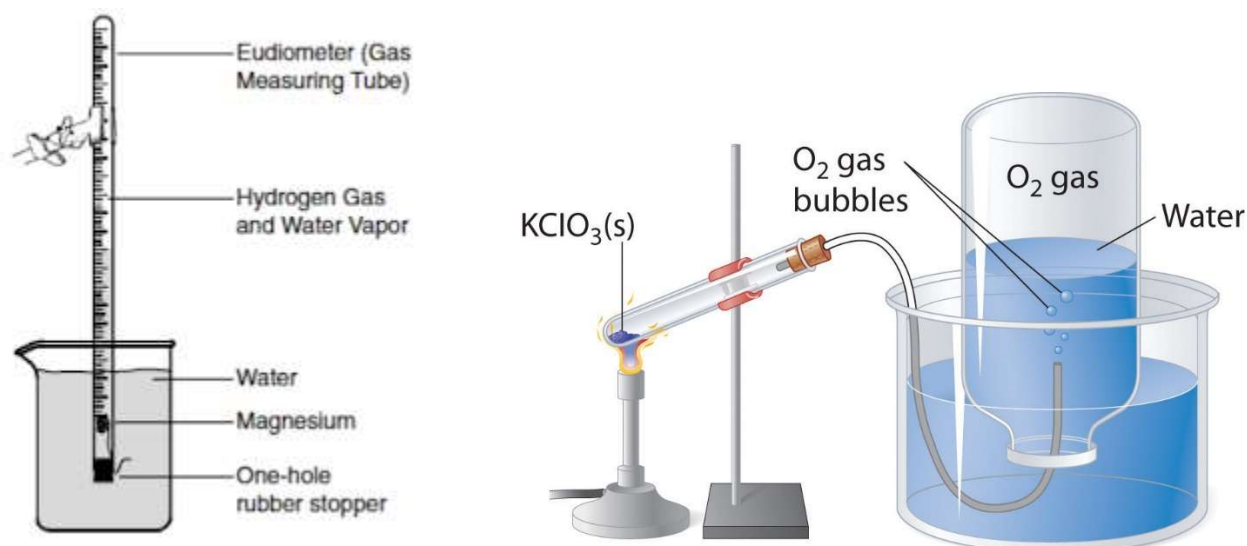
- Making solutions to dissolve substances for analysis, particularly in titrations.

## Important to Remember:

- Molarity = moles solute/L of solution

# Quick Guide to Experimental Procedures

## Gas Collection Over Water



## Common Applications:

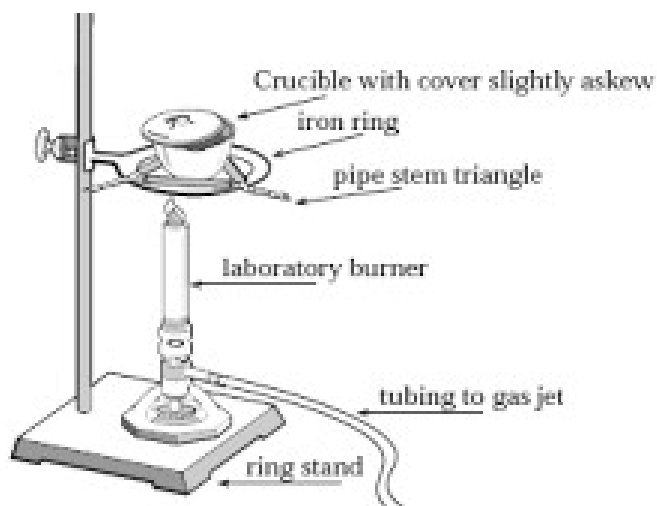
- Collecting gases that form in reactions like  
 $\text{Mg} + 2\text{HCl} \rightarrow \text{MgCl}_2 + \text{H}_2(\text{g})$   
 $2\text{KClO}_3 \rightarrow 2\text{KCl} + 3\text{O}_2(\text{g})$

## 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:

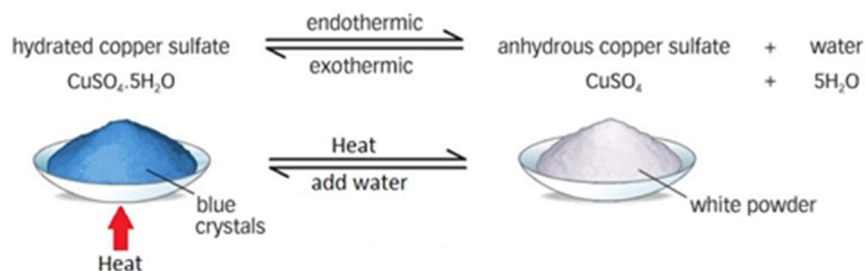
- **Not heating the hydrate enough**
  - Ratio of anhydrous salt: water will not be accurate, as water will remain in the sample
  - Appears fewer moles of water and more moles of salt will be in the hydrate
- **Overheating the hydrate**
  - Anhydrous salt could decompose in the heat
  - It will appear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

<https://socratic.org/questions/5504d4ee581e2a272ba7d3c8>

**Crucible Set Up:** [https://whs.rocklinusd.org/documents/Science/Epsom\\_salt\\_lab.pdf](https://whs.rocklinusd.org/documents/Science/Epsom_salt_lab.pdf)

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