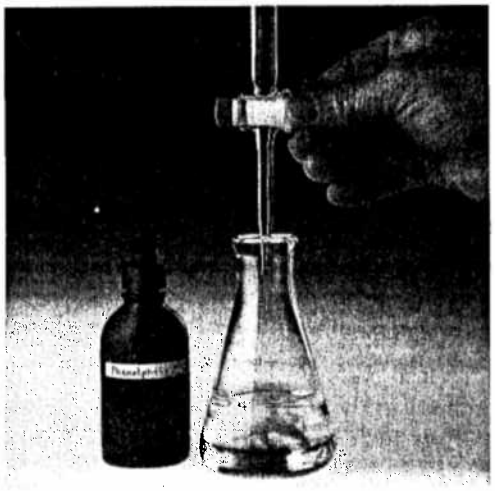


# Experiment 9

# A Volumetric Analysis

A titrimetric analysis requires the careful addition of titrant.

## OBJECTIVES

- To prepare and standardize a sodium hydroxide solution
- To determine the molar concentration of a strong acid

## TECHNIQUES

The following techniques are used in the Experimental Procedure


## INTRODUCTION

A chemical analysis that is performed primarily with the aid of volumetric glassware (e.g., pipets, burets, volumetric flasks) is called a **volumetric analysis.** For a volumetric analysis procedure, a known quantity or a carefully measured amount of one substance reacts with a to-be-determined amount of another substance with the reaction occurring in aqueous solution. The volumes of all solutions are carefully measured with volumetric glassware.

The known amount of the substance for an analysis is generally measured and available in two ways:

1. As a **primary standard:** An accurate mass (and thus, moles) of a solid substance is measured on a balance, dissolved in water, and then reacted with the substance being analyzed.
2. As a **standard solution:** A measured number of moles of substance is present in a measured volume of solution—a solution of known concentration, generally expressed as the molar concentration (or molarity) of the substance. A measured volume of the standard solution then reacts with the substance being analyzed.

*Primary standard: a substance that has a known high degree of purity, a relatively large molar mass, is nonhygroscopic, and reacts in a predictable way*

*Standard solution: a solution having a very well known concentration of a solute*

The reaction of the known substance with the substance to be analyzed, occurring in aqueous solution, is generally conducted by a titration procedure.

The titration procedure requires a buret to dispense a liquid, called the **titrant,** into a flask containing the **analyte** (Figure 9.1*a*). The titrant may be a solution of known or unknown concentration. The analyte may be a solution whose volume is measured with a pipet or it may be a dissolved solid with a very accurately measured mass. For the acid–base titration studied in this experiment, the titrant is a standard solution of sodium hydroxide and the analyte is an acid.

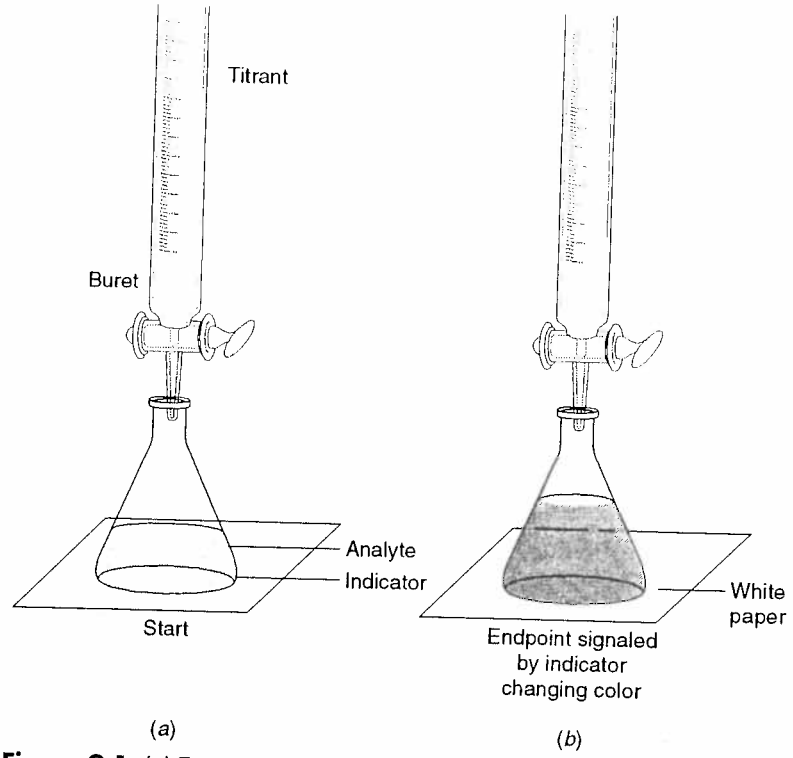



**Figure 9.1** (a) Titrant in the buret is dispensed into the analyte until (b) the indicator changes color at its endpoint.

*Stoichiometric amounts: amounts corresponding to the mole ratio of the balanced equation*

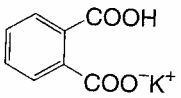
*Acid–base indicator: a substance having an acidic structure with a different color than its basic structure*

*pH: the negative logarithm of the molar concentration of $H_3O^+$, $pH = -\log[H_3O^+]$. Refer to Experiment 6.*

A reaction is complete when **stoichiometric amounts** of the reacting substances are combined. In a titration this is the **stoichiometric point.**[1] In this experiment the stoichiometric point for the acid–base titration is detected using a phenolphthalein **indicator.** Phenolphthalein is colorless in an acidic solution but pink in a basic solution. The point in the titration at which the phenolphthalein changes color is called the **endpoint** of the indicator (Figure 9.1*b*). Indicators are selected so that the stoichiometric point in the titration coincides (at approximately the same **pH**) with the endpoint of the indicator.

## Standardization of a Sodium Hydroxide Solution

*Hygroscopic: able to absorb water vapor readily*




potassium hydrogen phthlate

Solid sodium hydroxide is very **hygroscopic;** therefore its mass cannot be measured to prepare a solution with an accurately-known molar concentration (a primary standard solution). To prepare a NaOH solution with a very well known molar concentration, it must be standardized with an acid that *is* a primary standard.

In Part A of this experiment, *dry* potassium hydrogen phthalate, $KHC_8H_4O_4$, is used as the primary acid standard for determining the molar concentration of a sodium hydroxide solution. Potassium hydrogen phthalate is a white, crystalline, acidic solid. It has the properties of a primary standard because of its high purity, relatively high molar mass, and because it is only *very slightly* hygroscopic. The moles of $KHC_8H_4O_4$ used for the analysis is calculated from its measured mass and molar mass (204.44 g/mol):

$$\text{mass } (g)\ KHC_8H_4O_4 \times \frac{\text{mol } KHC_8H_4O_4}{204.44 \text{ g } KHC_8H_4O_4} = \text{mol } KHC_8H_4O_4 \qquad (9.1)$$

From the balanced equation for the reaction, one mole of $KHC_8H_4O_4$ reacts with one mole of NaOH according to the equation:

$$KHC_8H_4O_4(aq) + NaOH(aq) \rightarrow H_2O(l) + NaKC_8H_4O_4(aq) \qquad (9.2)$$

[1]The stoichiometric point is also called the **equivalence point**, indicating the point at which stoichiometrically equivalent quantities of the reacting substances are combined.

In the experimental procedure an accurately measured mass of dry potassium hydrogen phthalate is dissolved in deionized water. A prepared NaOH solution is then dispensed from a buret into the $KHC_8H_4O_4$ solution until the stoichiometric point is reached, signaled by the colorless to pink change of the phenolphthalein indicator. At this point the dispensed volume of NaOH is noted and recorded.

The molar concentration of the NaOH solution is calculated by determining the number of moles of NaOH used in the reaction (Equation 9.2) and the volume of NaOH dispensed from the buret.

$$\text{molar concentration } (M) \text{ of NaOH (mol/L)} = \frac{\text{mol NaOH}}{\text{L of NaOH solution}} \qquad (9.3)$$

Once the molar concentration of the sodium hydroxide is calculated, the solution is said to be "standardized" and the sodium hydroxide solution is called a **secondary standard** solution.

### Molar Concentration of an Acid Solution

In Part B, an unknown molar concentration of an acid solution is determined. The standardized NaOH solution is used to titrate an accurately measured volume of the acid to the stoichiometric point. By knowing the volume and molar concentration of the NaOH, the number of moles of NaOH used for the analysis is

$$\text{volume (L)} \times \text{molar concentration (mol/L)} = \text{mol NaOH} \qquad (9.4)$$

From the stoichiometry of the reaction, the moles of acid neutralized in the reaction can be calculated. If your acid of unknown concentration is a monoprotic acid, HA (as is HCl(*aq*)), then the mole ratio of acid to NaOH will be 1:1 (Equation 9.5). However, if your acid is diprotic, $H_2A$ (as is $H_2SO_4$), then the mole ratio of acid to NaOH will be 1:2 (Equation 9.6). Your instructor will inform you of the acid type, HA or $H_2A$.

$$HA(aq) + NaOH(aq) \rightarrow NaA(aq) + 2H_2O(l) \qquad (9.5)$$

$$H_2A(aq) + 2NaOH(aq) \rightarrow Na_2A(aq) + 2H_2O(l) \qquad (9.6)$$

From the moles of the acid that react and its measured volume, the molar concentration of the acid is calculated:

$$\text{molar concentration of the acid (mol/L)} = \frac{\text{mol acid}}{\text{volume of acid (L)}} \qquad (9.7)$$

## EXPERIMENTAL PROCEDURE

**Procedure Overview:** A NaOH solution is prepared with an approximate concentration. A more accurate molar concentration of the NaOH solution (as the titrant) is determined using dry potassium hydrogen phthalate as a primary standard. The NaOH solution, now a secondary standard solution, is then used to determine the "unknown" molar concentration of an acid solution.

### A. The Standardization of a Sodium Hydroxide Solution



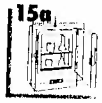
You are to complete at least three "good" trials (±1% reproducibility) in standardizing the NaOH solution. Prepare three clean 125-mL or 250-mL Erlenmeyer flasks for the titration.

You will need to use approximately one liter of boiled, deionized water for this experiment. Start preparing that first.

1. **Prepare the Stock NaOH Solution.**[2] One week before the scheduled laboratory period, dissolve about 4 g of NaOH (pellets or flakes) (**Caution:** *NaOH is very corrosive—do not allow skin contact. Wash hands thoroughly with water.*) in 5 mL of deionized water in a 150-mm rubber-stoppered test tube. Thoroughly

[2]Check with your laboratory instructor to see if the NaOH solution is prepared for Part A.1 (and/or Part A.3) and to see if the $KHC_8H_4O_4$ is dried for Part A.2.

mix and allow the solution to stand for the precipitation of sodium carbonate, $Na_2CO_3$.[3]



2. **Dry the Primary Standard Acid.** Dry 2–3 g of $KHC_8H_4O_4$ at 110°C for several hours in a constant temperature drying oven. Cool the sample in a desiccator.

3. **Prepare the Diluted NaOH Solution.** Decant about 4 mL of the NaOH solution prepared in Part A.1 into a 500-mL polyethylene bottle (Figure 9.2). (**Caution:** *Concentrated NaOH solution is extremely corrosive and will cause severe skin removal!*) Dilute to 500 mL with previously boiled,[4] deionized water cooled to room temperature. Cap the polyethylene bottle to prevent the absorption of $CO_2$. Swirl the solution and label the bottle.

   Calculate an *approximate* molar concentration of your diluted NaOH solution.


*Tared mass: mass of a sample without regard to its container*

4. **Prepare the Primary Standard Acid.** a. Calculate the mass of $KHC_8H_4O_4$ that will require about 15–20 mL of your diluted NaOH solution to reach the stoichiometric point. Show the calculations on the Report Sheet.

   b. Measure this mass (±0.001 g) of $KHC_8H_4O_4$ on a **tared** piece of weighing paper (Figure 9.3) and transfer it to a clean, labeled Erlenmeyer flask. Similarly, prepare all three samples while you are occupying the balance. Dissolve the $KHC_8H_4O_4$ in about 50 mL of previously boiled, deionized water and add 2 drops of phenolphthalein.

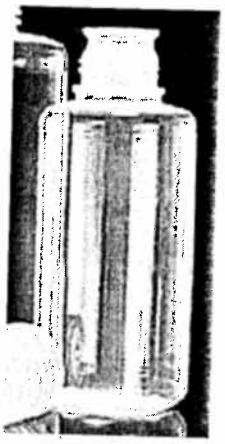


**Figure 9.2** A 500-mL polyethylene bottle for the NaOH solution.

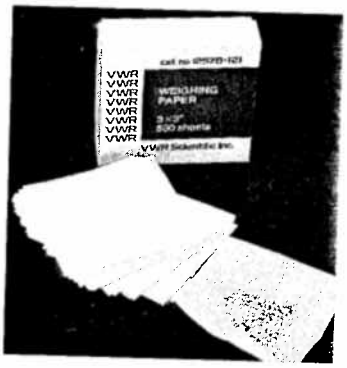



**Figure 9.3** Weighing paper for the $KHC_8H_4O_4$ measurements.



5. **Prepare a Clean Buret.** Wash a 50-mL buret and funnel thoroughly with soap and water using a long buret brush. Flush the buret with tap water and rinse several times with deionized water. Rinse the buret with three 5-mL portions of the diluted NaOH solution, making certain that the solution wets the entire inner surface. Drain each rinse through the buret tip. Discard each rinse in the "Waste Bases" container. Have the instructor approve your buret and titration setup before continuing.

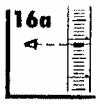
---

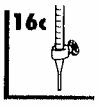
[3]Carbon dioxide, $CO_2$, from the atmosphere is an **acidic anhydride** (meaning that when $CO_2$ dissolves in water, it forms an acidic solution). The acid $CO_2$ reacts with the base NaOH to form the less soluble salt, $Na_2CO_3$.

$$CO_2(g) + 2NaOH(aq) \rightarrow Na_2CO_3(s) + H_2O(l)$$

[4]Boiling the water removes traces of $CO_2$ that would react with the sodium hydroxide in solution.

6. **Fill the Buret.** Using a clean funnel, fill the buret with the NaOH solution.[5] After 10–15 seconds, read the volume by viewing the bottom of the meniscus with the aid of a black line drawn on a white card (the buret can be removed from the stand or moved up or down in the buret clamp to make this reading; you need not stand on a lab stool to read the meniscus). Record this initial volume according to the guideline in Technique 16A.2, "using all certain digits (from the labeled calibration marks on the glassware) *plus* one uncertain digit (the last digit which is the best estimate between the calibration marks)." Place a sheet of white paper beneath the Erlenmeyer flask.




7. **Titrate the Primary Standard Acid #1.** Slowly add the NaOH titrant to the first acid sample prepared in Part A.4. Swirl the flask (with the proper hand[6]) after each addition. Initially, add the NaOH solution in 1- to 2-mL increments. As the stoichiometric point nears, the color fade of the indicator occurs more slowly. Occasionally rinse the wall of the flask with (previously boiled, deionized) water from your wash bottle. Continue addition of the NaOH titrant until the endpoint is reached. *The endpoint in the titration should be within one-half drop of a slight pink color* (see opening photo). The color should persist for 30 seconds. After 10–15 seconds, read (Figure 9.4) and record the final volume of NaOH in the buret.

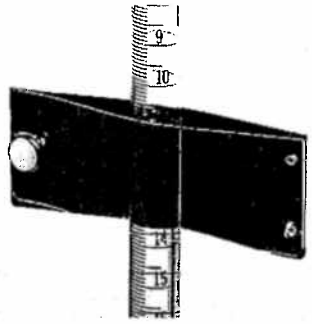


**Figure 9.4** Read the volume of titrant with a black background.

8. **Repeat the Analysis with the Remaining Standard Acid Samples.** Refill the buret and repeat the titration *at least* two more times with varying, but accurately known, masses of $KHC_8H_4O_4$.
9. **Do the Calculations.** Calculate the molar concentration of the diluted NaOH solution. The molar concentrations of the NaOH solution from the three analyses should be within ±1%. Place a corresponding label on the 500-mL polyethylene bottle.

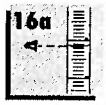
*Disposal:* Dispose of the neutralized solutions in the Erlenmeyer flasks in the "Waste Acids" container.


## B. Molar Concentration of an Acid Solution

Three samples of the acid having an unknown concentration are to be analyzed. Ask your instructor for the acid type of your unknown (i.e., HA or $H_2A$). Prepare three *clean* 125- or 250-mL Erlenmeyer flasks for this determination.



1. **Prepare the Acid Samples of Unknown Concentration.** In an Erlenmeyer flask, pipet 25.00 mL of the acid solution. Add 2 drops of phenolphthalein.
2. **Fill the Buret and Titrate.** Refill the buret with the (now) standardized NaOH solution and, after 10–15 seconds, read and record the initial volume. Refer to Parts A.6 and A.7. Titrate the acid sample to the phenolphthalein endpoint. Read and record the final volume of titrant.





3. **Repeat.** Similarly titrate the other samples of the acid solution.
4. **Calculations.** Calculate the average molar concentration of your acid unknown.

**Save.** Save your standardized NaOH solution in the *tightly capped* 500-mL polyethylene bottle for Experiments 10, 17, 18, and/or 19. Consult with your instructor.

*Disposal:* Dispose of the neutralized solutions in the "Waste Acids" container. Consult with your instructor.


[5]Be certain all air bubbles are removed from the buret tip.
[6]Check Technique 16C.3 for this procedure.

**CLEANUP:** Rinse the buret and pipet several times with tap water and discard through the tip into the sink. Rinse twice with deionized water. Similarly clean the Erlenmeyer flasks.

Check and clean the balance area. All solids should be discarded in the "Waste Solid Acids" container.

**The Next Step**

What are the acid concentrations for various noncarbonated soft drinks? the acid of vinegar (Experiment 10), the acids used for treating swimming pools? the acid of fruit juices? the antacids (Experiment 17), of aspirin (Experiment 19), . . . specifically, what are those acids? Design a procedure for determining the acidity for a select grouping of foods, drinks, or other familiar commercial products.

## NOTES AND CALCULATIONS

# Experiment 9 *Prelaboratory Assignment*

## A Volumetric Analysis

Date _____ Lab Sec. _____ Name _____ Desk No. _____

1. a. Define the analyte in a titration.

   b. Is the indicator generally added to the titrant or the analyte in a titration?

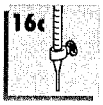
2. a. What is the primary standard used in this experiment (name and formula)? Define a primary standard.

   b. What is the secondary standard used in this experiment (name and formula)? Define a secondary standard.

3. Distinguish between a stoichiometric point and an endpoint in an acid–base titration.

4. a. How do you know that glassware (e.g., a buret or pipet) is clean?


   b. When rinsing a buret after cleaning it with soap and water, should the rinse be dispensed through the buret tip or the top opening of the buret? Explain.




   c. Experimental Procedure, Part A.5. In preparing the buret for titration the final rinse is with the NaOH titrant rather than with deionized water. Explain.

   d. Experimental Procedure, Part A.7. How is a "half-drop" of titrant dispensed from a buret?

5. Experimental Procedure, Part A.1. A 4-g mass of NaOH is dissolved in 5 mL of water.
   a What is the approximate molar concentration of the NaOH?

   b. In Part A.3, a 4-mL aliquot of this solution is diluted to 500 mL of solution. What is the approximate molar concentration of NaOH in the diluted solution? Enter this information on your Report Sheet.

   c. Part A.4. Calculate the mass of $KHC_8H_4O_4$ (molar mass = 204.44 g/mol) that reacts with 15 mL of the NaOH solution in Part A.3.

6. a. A 0.4040-g sample of potassium hydrogen phthalate, $KHC_8H_4O_4$ (molar mass = 204.44 g/mol) is dissolved with 50 mL of deionized water in a 125-mL Erlenmeyer flask. The sample is titrated to the phenolphthalein endpoint with 14.71 mL of a sodium hydroxide solution. What is the molar concentration of the NaOH solution?

   b. A 25.00-mL aliquot of a nitric acid solution of unknown concentration is pipetted into a 125-mL Erlenmeyer flask and 2 drops of phenolphthalein are added. The *above* sodium hydroxide solution (the titrant) is used to titrate the nitric acid solution (the analyte). If 18.92 mL of the titrant is dispensed from a buret in causing a color change of the phenolphthalein, what is the molar concentration of the nitric acid solution?

# Experiment 9 *Report Sheet*

# A Volumetric Analysis

Date _____ Lab Sec. _____ Name _____ Desk No. _____

Maintain at least three significant figures when recording data and performing calculations.

**A. Standardization of a Sodium Hydroxide Solution**

Calculate the approximate molar concentration of diluted NaOH solution (Part A.3).

Calculate the approximate mass of $KHC_8H_4O_4$ for the standardization of the NaOH solution (Part A.4).

| | *Trial 1* | *Trial 2* | *Trial 3* |
|---|---|---|---|
| 1. Tared mass of $KHC_8H_4O_4$ (g) | | | |
| 2. Molar mass of $KHC_8H_4O_4$ | | 204.44 g/mol | |
| 3. Moles of $KHC_8H_4O_4$ (mol) | | | |
| Titration apparatus approval | | | |
| 4. Buret reading of NaOH, *initial* (mL) | | | |
| 5. Buret reading of NaOH, *final* (mL) | | | |
| 6. Volume of NaOH dispensed (mL) | | | |
| 7. Molar concentration of NaOH (mol/L) | | | |
| 8. Average molar concentration of NaOH (mol/L) | | | |
| 9. Standard deviation of molar concentration | | | |
| 10. Relative standard deviation of molar concentration (%RSD) | | | |

### B. Molar Concentration of an Acid Solution

Acid type: _____ Unknown No. _____

Balanced equation for neutralization of acid with NaOH.

| | *Sample 1* | *Sample 2* | *Sample 3* |
|---|---|---|---|
| 1. Volume of acid solution (mL) | 25.0 | 25.0 | 25.0 |
| 2. Buret reading of NaOH, *initial* (mL) | | | |
| 3. Buret reading of NaOH, *final* (mL) | | | |
| 4. Volume of NaOH dispensed (mL) | | | |
| 5. Molar concentration of NaOH (mol/L), Part A | | | |
| 6. Moles of NaOH dispensed (mol) | | | |
| 7. Molar concentration of acid solution (mol/L) | | | |
| 8. Average molar concentration of acid solution (mol/L) | | | |
| 9. Standard deviation of molar concentration | | | |
| 10. Relative standard deviation of molar concentration (%RSD) | | | |

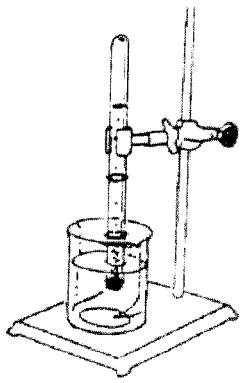
### Laboratory Questions

Circle the questions that have been assigned.

1. Part A.2. Pure potassium hydrogen phthalate is used for the standardization of the sodium hydroxide solution. Suppose that the potassium hydrogen phthalate is *not* completely dry. Will the reported molar concentration of the sodium hydroxide solution be too high, too low, or unaffected because of the moistness of the potassium hydrogen phthalate? Explain.
2. Part A.3. The student "forgot" to prepare any boiled, deionized water for the preparation of the NaOH solution and *then* "forgot" to cap the bottle. Will the concentration of the NaOH solution be greater than, less than, or unaffected by this carelessness? Explain.
3. Part A.4. Phenolphthalein is a weak organic acid, being colorless in an acidic solution and pink in a basic solution. The Experimental Procedure suggests the addition of 2 drops of phenolphthalein for the standardization of the sodium hydroxide solution. Explain why the analysis will be less accurate with the addition of a larger amount, e.g., 20 drops, of phenolphthalein.
4. Part A.7. A drop of the NaOH titrant adheres to the side of the buret (because of a dirty buret) between the initial and final readings for the titration. How does this "clean glass" error affect the reported molar concentration of the NaOH solution? Explain.
5. Part B.2. The wall of the Erlenmeyer flask is occasionally rinsed with water from the wash bottle (see Part A.7) during the analysis of the acid solution. How does this affect the reported molar concentration of the acid solution? Explain.
6. Parts A.7 and B.2. For the standardization of the NaOH solution in Part A.7, the endpoint was consistently reproduced to a dark pink color. However, the endpoint for the titration of the acid solution in Part B.2 was consistently reproduced to a faint pink color. Will the reported molar concentration of the acid solution be too high, too low, or unaffected by the differences in the colors of the endpoints. Explain.

Name: _____ Block: ___ Date: _____

# *Gas Laws Lab*



## Purpose

In this experiment, you will produce hydrogen gas, $H_2$,by reacting magnesium with hydrochloric acid according to the following equation:

$$Mg + 2\ HCl \rightarrow MgCl_2 + H_2$$

You will assume the hydrogen is an ideal gas, and you will measure its mass, volume, temperature, and pressure. From these measured values, you will calculate the molar volume of hydrogen and compare the result with the ideal value above.

## Materials

Eudiometer (Buret)
Hydrochloric acid (6 M)
Magnesium ribbon
Copper wire
Thermometer

### APPLICATION OF PRINCIPLES

1. Tell whether the following errors would **increase**, **decrease**, or have **no effect** on the experimental molar volume.

   a) the measured mass of the magnesium was too small

   b) the actual temperature of the hydrogen is less than room temperature.

2. If you obtain 3.0 cm of magnesium ribbon predict the theoretical volume of hydrogen gas produced. (Mass of magnesium ribbon is 0.871g/meter)

## Procedure:

1. Measure a piece of magnesium ribbon 3.0 cm long. Do not exceed 3.0 cm. This strip has been pre-measured so that it will not produce more hydrogen than the collection tube will hold.

2. Determine the mass by using the conversion factor of 0.871 g/m of magnesium ribbon.

3. Produce and collect the hydrogen gas as follows: Claim a gas collection tube (already set up in the lab). Fold up the magnesium ribbon into a small, tight bundle. Tie it with a piece of thread that is 10 to 15 *cm* long.

4. Add about 10 *mL* of 6.0M HCl to the gas collection tube. **CAUTION**. *Be sure to use* ***hydrochloric*** *acid; others might react violently when the water is added.* Then fill the tube completely with tap water, until it is nearly overflowing.

5. Place the magnesium in the mouth of the tube so that it is about 3 *cm* below the surface of the water. Fold the thread extension over the side of the tube. Insert a one- or two-hole stopper into the opening so that the cage is held firmly in place.

6. Holding your finger over the stopper hole(s), invert the tube into a 400 *mL* beaker that is about half-filled with water. Then clamp the tube in place as shown in the diagram, with its mouth below the water's surface. (There is no need to rush this maneuver. The acid will take more than a minute to diffuse down to the stopper, and by then it becomes dilute enough not to harm your finger.

7. Observe the reaction. When no more hydrogen bubbles are visible, the reaction is complete. Wait an additional 5 minutes so that the hydrogen gas comes to room temperature.

8. Cover the hole in the stopper with your finger and transfer the tube to a large cylinder or battery jar filled with water. Lower or raise the tube until the liquid level on the inside of the tube is the same as the outside. Record the volume of the hydrogen gas. (Be sure to read the liquid level at eye level.)

9. Record room temperature. Obtain the vapor pressure of water from the table. Record.

| Temperature (°C) | Vapor Pressure (mmHg) |
|---|---|
| 18 | 15.0 |
| 19 | 16.5 |
| 20 | 17.5 |
| 21 | 18.7 |
| 22 | 19.8 |
| 23 | 21.1 |
| 24 | 22.4 |
| 25 | 23.8 |
| 26 | 25.2 |
| 27 | 26.7 |
| 28 | 28.3 |
| 29 | 30.0 |

10. To find the partial pressure exerted by the hydrogen, you must recognize that the atmospheric pressure equals the partial pressure of hydrogen gas in the tube (when the water levels are equal), plus the partial pressure of water vapor mixed with the

hydrogen (according to the equation below).

$$P_{atm} = P_{H2} + P_{H2O}$$

11. Record today's atmospheric pressure and temperature.

12. Using the combined gas law equation (shown below), convert your measured volume of hydrogen (from Step 8) to conditions of STP, 1 atm pressure and 0 °C (273 K). Give the result in units of liters (*L*).

$$P_1 V_1 / T_1 \text{ (today's conditions)} = P_2 V_2 / T_2 \text{ (STP)}$$

13. Compare your experimental hydrogen volume (adjusted for partial pressure of water and current pressure and temperature) to the theoretical value by calculating the percent yield.

## Data Table

| | |
|---|---|
| **1. Mass of magnesium strip** | |
| **2. Room temperature ($T_1$)** | |
| **3. Atmospheric pressure** | |
| **4. Volume of the hydrogen ($V_1$)** | |
| **5. Partial pressure of the water** | |
| **6. Partial pressure of hydrogen gas ($P_1$)** | |
| **7. Volume of Hydrogen collected, adjusted to STP ($V_2$)** | |
| **8. Standard temperature ($T_2$)** | |
| **9. Standard Pressure ($P_2$)** | |
| **10. Percent Yield** | |

**CALCULATIONS:**

**DISCUSSION:**

1. **There are other alternative ways to determine the theoretical volume of hydrogen production.**

   **PV = nRT is another option. Because the molar ratio of magnesium to hydrogen gas is 1 to 1, if you know the number of moles of magnesium that reacted, you also know the number of moles of hydrogen gas.**

   a. **Mass of magnesium:** _____

   b. **Moles of magnesium:** _____

   c. **Moles of hydrogen gas:** _____

   d. **Volume of hydrogen gas:** _____ **(Theoretical yield)**

2. **Discuss at least two reason that you had an actual yield that did not match the theoretical yield.**

**Name**_____ **Block:** ___ **Date:** _____

**Analytical Chemistry – Gravimetric Lab**

## Purpose:

To calculate the carbon content of a marshmallow by thermo-gravimetric means. The amount of carbon will be determined by using the energy of combustion to first drive off the water content and next to reduce the carbohydrates to carbon. The amount of carbon will be directly measured by massing the ashes remaining after the experiment. The theoretical percent of carbon in a marshmallow will be calculated based on the ratio of $CH_2O$ in a carbohydrate and the mass of carbohydrates vs. the total mass of the marshmallow.

## Materials:

evaporating dish
Bunsen burner
Analytical scale
wire guaze
flint striker
three samples of mini-marshmallows
crucible tongs

## Pre-lab Activity:

**Nutrition Facts**

Serving Size 50 g

| Amount Per Serving | |
|---|---|
| Calories 159 | Calories from Fat 1 |
| | % Daily Value* |
| Total Fat 0g | 0% |
| Saturated Fat 0g | 0% |
| Trans Fat | |
| Cholesterol 0mg | 0% |
| Sodium 40mg | 2% |
| Total Carbohydrate 41g | 14% |
| Dietary Fiber 0g | 0% |
| Sugars 29g | |
| Protein 1g | |
| Vitamin A 0% • Vitamin C | 0% |
| Calcium 0% • Iron | 1% |

*Percent Daily Values are based on a 2,000 calorie diet. Your daily values may be higher or lower depending on your calorie needs.

NutritionData.com

1. Using the nutrition label to the left determine the percentage of a marshmallow that is carbohydrate.

2. Determine the percent carbon in a typical carbohydrate that is $CH_2O$.

3. Using the two percentages calculated in the previous questions determine the percentage of carbon in a marshmallow.

## Procedure:

1. Set up ring stand with iron ring clamp and wire gauze. Put evaporating dish on wire gauze. Position Bunsen burner under iron ring.

2. Using the analytical balance mass the evaporating dish. Record the mass on your data table.

3. Place the marshmallow in the evaporating dish and mass. Record the mass on your data table.

3. Light the Bunsen burner. Begin heating the substance. When the reaction stops (no more smoke) turn off Bunsen burner. Leave sample to cool.

4. Once the evaporating dish is cool enough to handle mass it and the remaining ashes. Record the mass on your data table.

5. Clean up evaporating dish and repeat steps 2-4 with the next two marshmallows.

6. Clean up: Place foods in the trash. Clean evaporating dish with steel wool if necessary. Return equipment.

## Discussion Questions:

1. What is considered the "signal" for a gravimetric analysis?
2. The iron content of an organometallic compound was determined by treating a 0.4873g sample with nitric acid and heating to volatilize the organic material. After ignition, the residue of Iron (III) oxide was 0.2091g. What is the %w/w of the iron in this compound?
3. For the question above what would happen to the %w/w if there was an incomplete combustion of the sample? (too high, too low, no change in comparison to the expected value) Explain your answer.
4. For the scenario in question #2 what would happen to the %w/w if the sample were heated too long and there was decomposition of the iron (III) oxide compound? (too high, too low, no change in comparison to the expected value) Explain your answer.

## Data Table:

| | Trial 1 | Trail 2 | Trail 3 |
|---|---|---|---|
| Mass of evaporating dish | g | g | g |
| Initial mass of dish and marshmallow | g | g | g |
| Final mass of dish and ashes | g | g | g |
| Mass of marshmallow | g | g | g |
| Mass of ashes | g | g | g |
| Percent ashes/marshmallow | % C | % C | % C |
| Average percent carbon | % C (experimental) | | |
| Theoretical percent carbon (from pre-lab) | % C (theoretical) | | |
| Percent Error | % Error | | |

## Calculations, Data Analysis and Conclusion:

Please record in your lab notebook.

Team Members: _____ Block: ___ Date: _____

**Investigation of Color and Absorbance**

**Purpose:**

The purpose of this lab is to introduce the concept of a spectrophotometer and provide understanding of how it works. Allowing students to practice scientific inquiry and discovery; determining the optimum wavelengths for absorption of the visible spectrum. Students will become familiar with Beer's Law and Planck's law and understand their application through graphical analysis (light intensity vs. wavelength).

**Materials:**

Food coloring (red, blue, green, yellow)
4 Pipettes
4 100 ml beakers
Lens paper
Glass stirring rod
50 ml Graduated cylinder
12 test tubes
cuvettes
Spec 20
Graphing paper

**Procedure: DAY 1**

**There are four team members so each team member is responsible for a separate color.**

1. Turn Spec 20 on to warm up lamp (left front knob). Set wavelength using the dial on the top of the Spec 20. View http://academics.wellesley.edu/Biology/Concepts/Html/analogspec20instructions.html to gain understanding of how to use the Spec 20.
2. Create 50 ml colored water solution for each color by adding 1 drop of food coloring into 50 ml of tap water in a 100 ml beaker. (one beaker for each color) This will be your 1:1 solution.
3. Prepare a BLANK cuvette by adding all solvents EXCEPT the substance to be measured (for this experiment the blank is water).

**A BLANK is used to calibrate the Spec 20 so that any absorbance attributable to the solvent and/or glass cuvette can be compensated. By zeroing the Spec 20 to the blank, you will measure *only* the absorbance due to the substance in question.**

4. With no tube in the holder, adjust the meter needle to read infinite absorbance (= 0% transmittance) using the ***left*** front knob (= power switch).
5. Using lens paper, wipe off/polish the outside of the BLANK cuvette to remove greasy finger smudges etc. (You might want to wear gloves). Using a wax pencil or Sharpie, make a small vertical mark at the top of each cuvette for alignment in the sample holder.
6. Raise the sample holder trapdoor and insert the cuvette such that the line on the cuvette lines up with the line on the sample holder. Close the lid.
7. Using the ***right*** front knob, adjust the meter needle to read absorbance = **0.0** (= 100 % transmittance). This step is called setting the "full scale".

- **Measuring Absorbance or Transmittance on the Spec 20**

8. Remove BLANK and insert cuvette containing your sample. Close lid.
9. Read the absorbance (lower scale) OR transmittance (upper scale) as appropriate for your sample.
10. Repeat for subsequent samples which use the *same* BLANK. (SEE NOTE BELOW)

NOTE: When taking several measurements at the same wavelength over a short time period, you do not need to reblank for each. Over longer times, however, the unit may drift and recalibration to the BLANK will be necessary. IF, however, you change the wavelength, you *must* re-zero the instrument. If you are taking readings over an extended period or sharing the instrument, re-zero for each measurement.

11. For each color determine the absorbance of the 1:1 solution at each of the ten prescribed wavelengths. Record your readings on the data tables provided.
12. Determine the optimum wavelength for each color. Verify results with teacher.
13. Turn off the Spec 20 and clean all cuvettes used, return cuvettes to the teacher.
14. Dispose of all solutions down the sink with plenty of water to rinse. Clean all equipment and return to its appropriate place.

**DATA TABLES:**

**Red Solution**

| | Absorbance Reading | | | | |
|---|---|---|---|---|---|
| **Wavelength** | **1:1 solution** | **1:2 solution** | **1:4 solution** | **1:8 solution** | **1:16 solution** |
| **400 nm** | | | | | |
| **425 nm** | | | | | |
| **450 nm** | | | | | |
| **475 nm** | | | | | |
| **500 nm** | | | | | |
| **525 nm** | | | | | |
| **550 nm** | | | | | |
| **575 nm** | | | | | |
| **600 nm** | | | | | |
| **625 nm** | | | | | |
| **650 nm** | | | | | |

Blue Solution

| | Absorbance Reading | | | | |
|---|---|---|---|---|---|
| Wavelength | 1:1 solution | 1:2 solution | 1:4 solution | 1:8 solution | 1:16 solution |
| 400 nm | | | | | |
| 425 nm | | | | | |
| 450 nm | | | | | |
| 475 nm | | | | | |
| 500 nm | | | | | |
| 525 nm | | | | | |
| 550 nm | | | | | |
| 575 nm | | | | | |
| 600 nm | | | | | |
| 625 nm | | | | | |
| 650 nm | | | | | |

Green Solution

| | Absorbance Reading | | | | |
|---|---|---|---|---|---|
| Wavelength | 1:1 solution | 1:2 solution | 1:4 solution | 1:8 solution | 1:16 solution |
| 400 nm | | | | | |
| 425 nm | | | | | |
| 450 nm | | | | | |
| 475 nm | | | | | |
| 500 nm | | | | | |
| 525 nm | | | | | |
| 550 nm | | | | | |
| 575 nm | | | | | |
| 600 nm | | | | | |
| 625 nm | | | | | |
| 650 nm | | | | | |

**Yellow Solution**

| | Absorbance Reading | | | | |
|---|---|---|---|---|---|
| **Wavelength** | **1:1 solution** | **1:2 solution** | **1:4 solution** | **1:8 solution** | **1:16 solution** |
| **400 nm** | | | | | |
| **425 nm** | | | | | |
| **450 nm** | | | | | |
| **475 nm** | | | | | |
| **500 nm** | | | | | |
| **525 nm** | | | | | |
| **550 nm** | | | | | |
| **575 nm** | | | | | |
| **600 nm** | | | | | |
| **625 nm** | | | | | |
| **650 nm** | | | | | |

**Procedure: DAY 2**

1. Create dilutions of each color by adding 4 ml of tap water to each of 4 test tubes for each color. Label the tubes as follows:

| | | | |
|---|---|---|---|
| Color: _____<br>1:2 dilution | Color: _____<br>1:4 dilution | Color: _____<br>1:8 dilution | Color: _____<br>1:16 dilution |

2. In the 1:2 test tube add 4 ml of the 1:1 color solution, mix well. Test the dilution at the optimum wavelength and record result.
3. Take 4 ml of the 1:2 dilution and add to the 1:4 test tube, mix well. Test the dilution at the optimum wavelength and record result.
4. Take 4 ml 1:4 dilution and add to the 1:8 test tube, mix well. Test the dilution at the optimum wavelength and record result.
5. Take 4 ml 1:8 dilution and add to the 1:16 test tube. Test the dilution at the optimum wavelength and record result.

   ****This process is called serial dilution.**
6. Turn off the Spec 20 and clean all cuvettes used, return cuvettes to the teacher.
7. Dispose of all solutions down the sink with plenty of water to rinse. Clean all equipment and return to its appropriate place.

**Data Analysis:**

1. Graph the 1:1 solution results on the same sheet of graphing paper using the appropriate colored pencil to create the line for the results. Be sure that all vertices are labeled and your graph has a title.
2. Graph the solution results for the dilutions of the individual colors, each color on its own graph.

**Discussion Questions:**

1. What was the purpose of the blank? For this lab you used tap water, why would it be important to have a blank that is created with the sample matrix minus the analyte being tested?

2. What did you notice about the absorbance readings for the 1:1 solutions of the different colors through the entire spectrum of wavelengths? List the optimum wavelengths for each color.

3. What did you notice about the graph of the individual colors at the set wavelengths for the variety of dilutions? What information can be determined from this type of graph?

4. What would you expect the absorbance to be for a 1:3 dilution of red at its optimum wavelength?

5. What would you expect the absorbance to be for a 1:32 dilution of green at its optimum wavelength?

# Experiment 35

# Spectrophotometric Metal Ion Analysis

The absorbance of light indicates the relative concentrations of a substance in solution.

## OBJECTIVES

- To use a spectrophotometer to measure the concentration of a metal ion
- To use graphing techniques for data analysis
- To learn of the adaptability of spectrophotometric analyses

## TECHNIQUES

The following techniques are used in the Experimental Procedure


## INTRODUCTION

Many transition metal cations have color, but in respective concentration ranges may also be considered hazardous wastes. Disposal of electroplating baths containing e.g., copper, nickel, or chromium ions cannot be simply discarded without some type of treatment. Often the treatment is precipitation, but also changes in oxidation states, acidification, complexation, or simple dilution procedures are used prior to disposal.

Because many transition metal ions do have color, their concentrations can be determined by a visible spectroscopic analysis. Those metal ions that do not have color may be analyzed by using ultraviolet radiation; however, a flame atomic absorption spectrophotometric (FAAS) analysis is by far the more common technique. The basic principles for either of the analyses are the same—standard solutions of the metal ion of interest are prepared, the absorption of each is determined to prepare a calibration curve. The absorption of the unknown is then determined and its concentration is determined by referring to the calibration curve.

Before beginning this experiment, read closely the Introduction to Experiment 34. An understanding of the absorbance and transmission of electromagnetic radiation through samples of varying concentrations in a spectrophotometer is imperative for an appreciation of the underlying chemical principles of this experiment.

Mixtures of ions can be problematic. The absorption spectrum for one ion may overlap and interfere with that of another ion. For that reason, FAAS or an induced couple plasma-atomic emission spectroscopy (ICP-AES) technique minimizes *some* of the interferences.

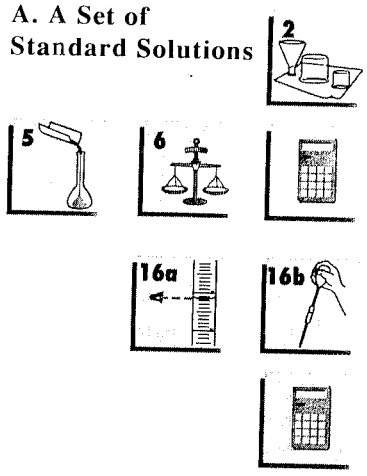
## EXPERIMENTAL PROCEDURE

**Procedure Overview:** A set of standard solutions is prepared. First, the wavelength for maximum absorption, $\lambda_{max}$, of the metal ion in the visible region is determined from an absorbance, *A*, versus $\lambda$ data plot. Next, the absorbance, *A*, of the standard solutions is determined at $\lambda_{max}$ in order to construct a calibration curve of *A* versus [*conc*]. The two data plots can be constructed with the use of suitable software (e.g.,

Excel). The concentration of the metal ion in the unknown sample is then determined from the calibration curve.

The metal ion for your analysis will be selected by the instructor (or chosen by you) from one of the following cations: $Cu^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Cr^{3+}$, or $Fe^{3+}$. You should also be aware that the color of some of these cations may be enhanced with the addition of a complexing agent. If a complexing agent is to be used in the analysis, it must be added to the stock solution in Part A and to the unknown in Part D. See Experiment 36. Consult your instructor.

## A. A Set of Standard Solutions




Identify the metal ion for analysis on the Report Sheet.

1. **Prepare a Stock Solution.** Use a clean 100-mL volumetric flask to prepare a 0.20 *M* solution of the metal ion selected for analysis using 0.1 *M* $HNO_3$ as a diluent.[1] To do this, you will need to know the formula of the salt and calculate the mass of the salt for the solution. See Prelaboratory Assignment, Exercise 2. The mass of the salt is to be measured $\pm 0.001$ g or as accurately as possible. Record on the Report Sheet the exact molar concentration of the stock solution.
2. **Prepare the Standard Solutions.** Prepare the solutions in Table 35.1 using clean, labeled 25-mL volumetric flasks (or 200-mm test tubes). Use pipets to dispense the solutions.

   Additional standard solutions may need to be prepared—the absorbance values for the set of standard solutions should range from 0 to ~1.1. See Part C.1. Calculate the molar concentrations of the standard solutions and record them in Part C of the Report Sheet.

## B. Determination of $\lambda_{max}$

1. **Calibrate the Spectrophotometer.** After the spectrophotometer has been turned on for at least 10 minutes, set the wavelength scale to its minimum (~350 nm), and set the zero (0%T) on the spectrophotometer. Rinse twice a cuvet with the blank solution. Fill the cuvet at least three-fourths full with the *blank solution* and dry the outside of the cuvet with a clean Kimwipe, removing fingerprints and water. Place the cuvet into the sample compartment, aligning the mark on the cuvet with that on the sample holder (or the clear sides of the cuvet in the path of EM radiation). The meter on the spectrophotometer should now read zero absorbance or 100%T. If not, consult with your instructor.

***Appendix B***

2. **Wavelength Scan.** Set the wavelength of the spectrophotometer to the shortest wavelength setting (~350 nm). Place the stock solution (*Solution 5* in Table 35.1) in a cuvet following the "blank solution procedure" in Part B.1. Measure and record the wavelength and absorbance.

   Scan the wavelength range of the spectrophotometer while recording absorbance values. The spectrophotometer may do this continuously/automatically or you may need to manually scan every ~10 nm. If you scan manually, set the

**Table 35.1** A Set of Standard Solutions for Metal Ion Analysis

| Standard Solution | 0.20 *M* Metal Ion | 0.1 *M* $HNO_3$ |
|---|---|---|
| Blank | 0 mL | dilute to 25 mL |
| 1 | 1 mL | dilute to 25 mL |
| 2 | 5 mL | dilute to 25 mL |
| 3 | 10 mL | dilute to 25 mL |
| 4 | 20 mL | dilute to 25 mL |
| 5 | 25 mL | — |

[1]Add the complexing agent, if applicable, prior to the dilution in the 100-mL volumetric flask. The amount of complexing agent can be determined from a literature search.

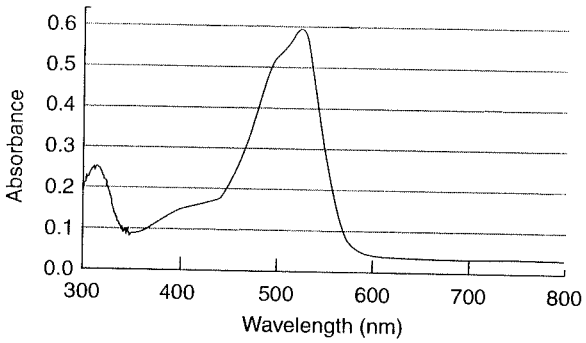



**Figure 35.1** A representative absorption spectrum of a laboratory solution. Provided by Mike Schuder, Carroll College, Minnesota.

absorbance at zero (100% T) with the blank solution (0.1 *M* $HNO_3$) at *each* chosen wavelength before recording the absorbance of the stock solution.

Plot the data, absorption, *A* (ordinate) versus wavelength, λ (abscissa), manually or with the appropriate software to determine the $\lambda_{max}$, the wavelength where maximum absorption occurs for your metal ion (see Figure 35.1). Ask your instructor to approve your graph.

## C. Plot the Calibration Curve

1. **Absorbance of Standard Solutions.** Set the spectrophotometer at $\lambda_{max}$. Record the absorbance of the stock solution (#5 in Table 35.1) at $\lambda_{max}$. Measure the absorbance for the other standard solutions in Table 35.1, starting with the most dilute (the blank).

   If *at least four* of the standard solutions are not in the absorbance range of 0 to ~1.1, prepare additional standard solutions.

2. **Plot the Data for the Calibration Curve.** Plot absorbance, *A* (ordinate), versus molar concentration (abscissa) for the six solutions in Table 35.1. Draw the best straight line through the data points to establish the calibration curve. Use Excel or similar graphing software to obtain values of the slope and y-intercept for the data plot. Ask your instructor to approve your graph.

***Appendix B***

## D. Unknown Metal Ion Concentration

1. **Prepare the Unknown.** Filter the solution if it is cloudy (for example, if it is a sample from an unknown source or sample from an electroplating bath).[2] Record the volume of the sample.[3] The solution may need to be diluted quantitatively (using 0.1 *M* $HNO_3$) with a pipet and 25-mL volumetric flask to have an absorbance measurement that falls within the range of the standard solutions (<1.1) on the calibration curve.
2. **Concentration of Metal Ion.** Once the sample is "prepared," measure its absorbance in the same manner as were the absorbance values for the standard solutions in Part C.1. Read the calibration curve to determine the molar concentration of the metal ion in the sample. Account for any dilution to determine the molar concentration of the metal ion in the original sample.
3. **Expressing Concentration.** Conventionally, in "real-world" samples, the metal ion concentrations are expressed in units of ppm (mg/L) or even smaller units depending upon the concentration of the metal. Express the concentration of your

[2]Turbidity in the sample can dramatically affect the absorbance reading of a sample and therefore affect the presumed concentration of the metal ion in solution.
[3]Add the complexing agent, if applicable, to unknown sample at this point.

metal ion "appropriately" in units of mass/volume. Mass per volume units may be parts per hundred (pph or %), parts per million (ppm), parts per billion (ppb), etc.


*Dispose of all prepared solutions in the "Waste Metal Salts" container.*

**The Next Step**

(1) Research the spectrophotometric analysis of a metal ion of interest. Design a procedure(s) for its analysis as a function of a few select parameters. (2) Read to determine the advantages (or disadvantages) of using FAAS or ICP-AES for the analysis of metal ions. What are the similarities in the procedure for FAAS and visible spectrophometric analyses? (3) Other metal ion analysis methods include the use of graphite furnace atomic absorption (GFAA), microwave induced plasma (MIP), and DC arc plasma . . . where are these methods of analysis most applicable?

# Spectrophotometric Metal Ion Analysis

Date _____ Lab Sec. _____ Name _____ Desk No. _____

1. Of the 100 mL of stock solution that is to be prepared for Part A.1, how many milliliters will be used for preparing the standard solutions in Part A.2?

2. A 100.0-mL volume of a 0.20 *M* stock solution of $Cu^{2+}$ is to be prepared using $CuSO_4 \cdot 5H_2O$ (molar mass = 249.68 g/mol).[2]
   a. How many grams of $CuSO_4 \cdot 5H_2O$ must be measured for the preparation?

   b. Describe the procedure for the preparation of the solution using 0.1 *M* $HNO_3$ as a diluent.

   c. A 2.0-mL pipet transfers the $Cu^{2+}$ stock solution to a 25.0-mL volumetric flask that is then diluted "to the mark" of the volumetric flask with 0.1 *M* $HNO_3$. What is the molar concentration of the diluted $Cu^{2+}$ solution?

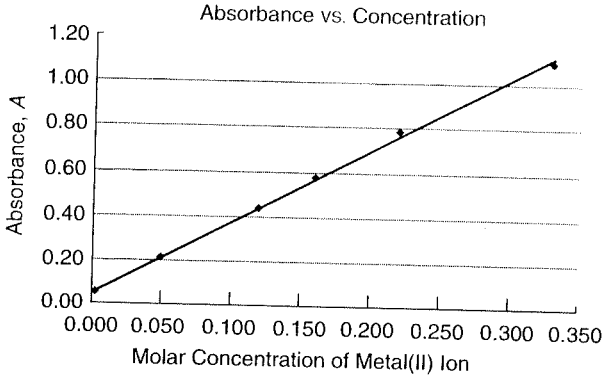
3. Refer to Figure 35.1.
   a. Identify the color of the visible spectrum where maximum absorption occurs (see Dry Lab 3).

   b. What is the predicted color of the solution?

   c. The $Co^{2+}$ ion has a $\lambda_{max}$ for absorption at 510 nm. Will a $Co^{2+}$ solution have the same exact color as the solution of Figure 35.1? Explain.

[2] $CuSO_4 \cdot 5H_2O$ is used as an agricultural fungicide, bactericide, and herbicide.

4. A calibration curve for a common metal(II) ion is shown:




a. A metal(II) ion solution of unknown concentration shows an absorbance of 0.71. What is the molar concentration of the metal(II) ion in the sample?

b. For a metal(II) ion solution having a 0.288 *M* concentration, what would be its predicted absorbance?

c. Assuming a 1-cm cell, what is the value of the absorptivity coefficient for this solution?

5. Briefly describe the procedure of setting $\lambda_{max}$ for a metal ion solution on the spectrophotometer.

6. The unknown metal ion concentration in your sample has an absorbance that is outside the range of the absorbance values for the standard solutions. What procedure(s) should be taken to rectify the discrepancy? Explain.

# Spectrophotometric Metal Ion Analysis

Date _____ Lab Sec. _____ Name _____ Desk No. _____

**Metal Ion for Analysis** _____

## A. A Set of Standard Solutions

1. **Prepare a Stock Solution.** Show the calculation for the mass of metal ion salt in the preparation of the stock solution.

Measured tared mass of metal ion salt (*g*) _____
Describe the preparation of the 0.20 *M* stock solution.

Concentration of stock solution (*mol/L*) _____

## B. Determination of $\lambda_{max}$

2. **Wavelength scan.** Use the following table to record wavelength/absorbance data.

| λ | Abs | λ | Abs | λ | Abs | λ | Abs | λ | Abs | λ | Abs | λ | Abs |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |

Plot the data of absorbance vs. wavelength to set $\lambda_{max}$. From the data plot, $\lambda_{max}$ = _____ nm

Have the instructor approve your graph. _____

## C. Plot the Calibration Curve

1. **Absorbance of Standard Solutions.** Read and record the absorbance values for the standard solutions.

| Standard Solution | Volume of Standard Solution (mL) | Absorbance | Calculated Molar Concentration |
|---|---|---|---|
| Blank | 0 | | |
| 1 | 1 | | |
| 2 | 5 | | |
| 3 | 10 | | |
| 4 | 20 | | |
| 5 | 25 | | |
| others as needed | | | |

Plot the calibration curve of absorbance vs. molar concentration for the standard solutions.

Calculate the absorptivity coefficient for the metal ion.

Have the instructor approve your graph. _____

**D. Unknown Metal Ion Concentration**

1. **Prepare the Unknown.** Describe the preparation for the solution containing the unknown concentration of the metal ion.

   Volume of unknown sample solution (*mL*) _____

   Volume of diluted sample (as needed) (*mL*) _____

2. **Concentration of Metal Ion.** From the calibration curve determine the molar concentration of the metal ion in the unknown solution.

   Concentration of metal ion from the calibration curve (*mol/L*) _____

   Concentration of metal ion in the original sample if corrected for dilution (*mol/L*) _____

   Show your calculations to account for the dilution of the original sample.

3. **Expressing Concentration.** Express the concentration of the metal ion in the sample in appropriate units of mass/volume, i.e., pph (%), ppm, ppb, etc.

   Concentration of metal ion in the original sample (*mass/volume*) _____

   Show calculations.

**Laboratory Questions**

Circle the questions that have been assigned.

1. Part A.1. For the preparation of the stock solution, 0.1 *M* $HNO_3$ is used as a diluent rather than deionized water. Explain why. Hint: Review the solubility rules of transition metal ions in Appendix G.
2. Part A.2. In diluting the standard solutions, 0.1 *M* $HNO_3$ is used. In the dilution, is it more important to use the correct volume or the correct concentration of the $HNO_3$ solution for the dilution? Explain.
3. Part B.2. Will $Co^{2+}(aq)$ or $Cu^{2+}(aq)$ have the shorter $\lambda_{max}$ for absorption in the visible spectrum? Explain. Hint: $Co^{2+}(aq)$ has a pink/rose color and $Cu^{2+}(aq)$ has a blue color. See Dry Lab 3.
4. Part D.1. An electrolytic-bath sample containing the metal ion of unknown concentration contains suspended matter. Because of a lack of time the absorbance of the sample was measured and recorded as is. As a result, will the concentration of the metal ion in the unknown be reported too high, too low, or remain unchanged as a result of chemist's hasty decision? Explain.
5. Part D.2. The cuvet used for the absorbance measurements is not wiped clean with a Kimwipe before the absorbance measurement. As a result, will the concentration of the metal ion in the unknown be reported too high, too low, or remain unchanged as a result of this poor technique? Explain.

# A TRILOGY LAB

BACKGROUND:

Benedict's reagent is used to determine the quantity of reducing sugars present in a sample. There are two types of Benedict's reagent, qualitative and quantitative, in this lab we will be comparing the two. Benedict's qualitative reagent contains copper (II) ions that can be reduced to copper (I) ions by reducing sugars such as glucose. When the blue copper (II) ions are reduced copper (I) oxide (a red precipitate) is formed. The quantity of reducing sugar present can be calculated by the change in saturation of the blue color of a mixture containing Benedict's reagent or by measuring the mass of the precipitate.


Benedict's quantitative reagent contains potassium thyocyanate and does not form red copper oxide. Instead the presence of reducing sugar is measured by the loss of the blue color of copper sulphate and a white/light green precipitate is formed which will settle out or can be removed by filtration before colorimetric determination of the filtrate.

Although the stoichiometry of the reaction is not precisely known, we do known that the stoichiometric proportions are 1 mole of glucose to 6.5 moles of copper (II). It has also been determined that 25ml of Benedict's reagent will react with 50 mg of glucose or 53mg of fructose. However, in this lab you will be performing a standardization to verify this information.

Both methods can be used to accurately measure the quantity of glucose in a solution or clinical specimen. In this lab you will use several different methods to determine the glucose concentration in a single unknown.

MATERIALS:

Qualitative Benedict's reagent
0.5% Glucose solution
1.0% Glucose solution
Unknown% Glucose solution
2-250 ml Erhlenmyer flask
Hot plate with stirring rod
6 pcs - Filter paper
Burette
Spectro-vis
Magnetic stirring rod

Quantitative Benedict's reagent
0.75% Glucose solution
1.5% Glucose solution
Anhydrous sodium carbonate
6-Test tubes
Boiling stones
Buchner funnel
Burette stand/clamp
Cuvettes

PROCEDURE (1):

1. Place a 400 ml beaker with 150ml of tap water and 2 boiling stones on a hot plate and bring to a boil.
2. Clearly label four clean 16x125 mm test tubes for the standards and two for the unknown.
3. Add 2.0 ml of each sample to the appropriate test tube.
4. Add 1.0 ml of qualitative Benedict's reagent to each test tube.
5. Mix the contents of each tube by gently shaking the test tubes back and forth.
6. Place the tubes in the beaker of boiling water. CAUTION! The water is very hot.
7. Incubate the tubes for 20 minutes. (during this time begin gathering materials and setting up for procedure two of this lab.)

8. Remove your test tubes and allow them to cool. (about 5 minutes, during this time hook up your Spectro-Vis to the lab Quest and calibrate with DI water.)
9. Record the color of the tubes on the data table.
10. Transfer the test tubes to the centrifuge and spin for two minutes. If there is still suspended debris in any of the tubes, centrifuge for two more minutes.
**It is important that the solution be clear for the absorbance measurements. If there is solid matter suspended in the solution, the light being sent through the sample will be scattered and will cause error in the measurements.
11. Decant the supernatants into clean, labeled test tubes. Be careful that any sediment remains in the pellet at the bottom of the test tube. Set the sediment aside for a different analysis (procedure 3).
12. Determine the percent absorbance of each of the samples using a Spectro-Vis, record the values from 735nm on the data table. **Remember to only touch the grated sides of the cuvettes!
13. Prepare a graph of the standard solutions and determine the concentration of each unknown.

PROCEDURE (2):

1. Set up the Buchner filtration apparatus and label 6 pieces of filter paper with your name and types of solution. **all filtrates can be collected in same Erlenmeyer flask, since it is waste material and can be washed down the drain.
2. Record the mass of the filter paper with pencil on the filter paper itself for each piece.
3. Rinse the precipitate from the 0.5% glucose solution (from procedure (1)) into the Buchner filtration apparatus with DI water. Be sure that all precipitate is rinsed onto the filter paper.
4. Run the filtration until all of the precipitate has been collected. Remove the filter paper and place in the oven for drying.
5. Repeat this procedure for all of the samples.
6. Allow the samples/filter papers to dry until the next class.
7. Mass the samples/filter paper, subtract the mass of the filter paper and record the mass of the precipitate on the data table.
8. Prepare a graph of the standard solutions and determine the concentration of each unknown.

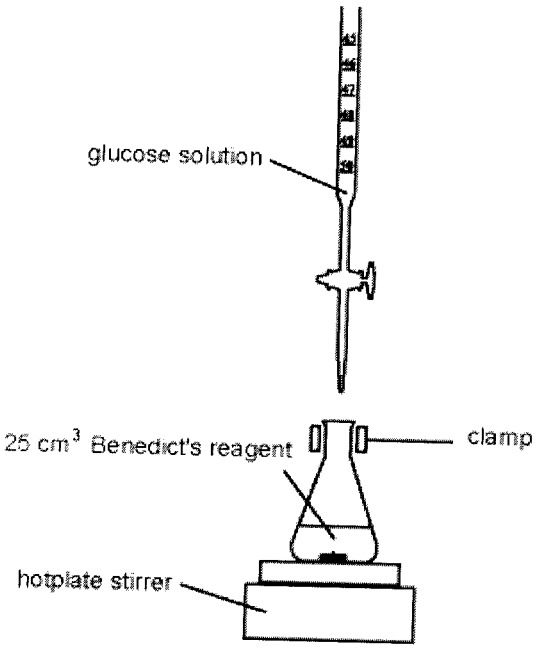
DATA TABLE 1

| Glucose Solution | Color of Tube | Absorbance at 735nm | Concentration of glucose mg/ml |
|---|---|---|---|
| 0.5% | | | |
| 0.75% | | | |
| 1.0% | | | |
| 1.5% | | | |
| Unk | | | |
| Unk | | | X |
| | | | X |

DATA TABLE 2

| Glucose Solution | Color of Precipitate | Mass of precipitate | Concentration of glucose mg/ml |
|---|---|---|---|
| 0.5% | | | |
| 0.75% | | | |
| 1.0% | | | |
| 1.5% | | | |
| Unk | | | |
| Unk | | | |

PROCEDURE (3):




1. Set up hot plate under a burette that has been clamped to a ring stand.
2. Add 50 ml of your 0.5% glucose standard to the burette.
3. Add 25ml of quantitative Benedict's reagent, 100ml of DI water and 10g of anhydrous sodium carbonate.
4. Add 2 boiling stones and stirring rod; bring mixture to a boil.
5. Once the mixture has dissolved begin adding the glucose solution, rapidly at first then dropwise when there is a change in the shade of blue color.
6. Continue adding glucose solution dropwise at 10-20 second intervals until the Benedict's solution turns clear. **The solution should remain boiling throughout the procedure, if it begins to dry out add more DI water. The precipitated copper (I) thiocyanate should be white or light green.
7. Once the trial first titration has been completed, run a second trial with the same glucose solution. In this trial run about 1ml less of the glucose solution than needed in the first run. Thereafter finish the titration dropwise, pausing a few second after each drop.
8. Record your result on the data table.
9. Repeat the procedure for each standard and twice for the unknown.
10. Prepare a graph of the standard solutions and determine the concentration of each unknown.

DATA TABLE 3

| Glucose Solution | Volume of Glucose Solution | Concentration of glucose mg/ml |
|---|---|---|
| 0.5% | | |
| 0.75% | | |
| 1.0% | | |
| 1.5% | | |
| Unk | | X |
| Unk | | X |

CALCULATIONS:

Using your graph determine the concentrations for the unknown glucose solutions, record the results here and then average the two results.

Result (1)

Unk1 _____ mg/ml Unk2 _____ mg/ml Average _____ mg/ml

Result (2)

Unk1 _____ mg/ml Unk2 _____ mg/ml Average _____ mg/ml

Result (3)

Unk1 _____ mg/ml Unk2 _____ mg/ml Average _____ mg/ml

DISCUSSION:

1. Could you create a semi-quantitative chart based on the colors formed during procedure (1)? Give an example chart that could be used based on your data.
2. How do your results compare from the three different procedures? If the actual value for the unknown was 0.9% create a standard deviation for your three results.
3. Calculate percent error for each of the procedures.
4. Evaluate the labs and analyze them for errors. As you discuss the errors, be sure that you indicate which procedure you are referring to.