

GEN CHEM LAB

MARY MCHALE

Gen Chem Lab



Gen Chem Lab

Collection edited by: Mary McHale

Content authors: Mary McHale, Alvin Orbaek, and Andrew Barron

Online: < <http://cnx.org/content/col10452/1.51> >

This selection and arrangement of content as a collection is copyrighted by Mary McHale.

It is licensed under the Creative Commons Attribution License:

<http://creativecommons.org/licenses/by/2.0/>

Collection structure revised: 2009/10/12

For copyright and attribution information for the modules contained in this collection, see the "[Attributions](#)" section at the end of the collection.

Gen Chem Lab

Table of Contents

[Chapter 1. Avogadro and All That](#)

[1.1.](#)

[Chapter 2. Stoichiometry: Laws to Moles to Molarity](#)

[2.1.](#)

[Experiment 3: Stoichiometry: Laws to Moles to Molarity](#)

[Objective](#)

[Grading](#)

[Before Coming to Lab...](#)

[Introduction](#)

[Titration Calculations:](#)

[Example:](#)

[Procedure](#)

[Materials List](#)

[Part 1](#)

[Materials List](#)

[Part 2](#)

[Chapter 3. Totally, Terrific Table](#)

[3.1.](#)

[Lab 4: Totally, Terrific Table!](#)

[Objective](#)

[Grading](#)

[Background Information](#)

[Introduction](#)

[Procedure](#)

[I. Activity Series](#)

[Part 1: Reactions of Metals with Water](#)

[Part 2: Reactions of Metals with HCl](#)

[CAUTION: This part of the procedure must be done in a fume hood!!](#)

[Part 3: Reactions of Metals with Other Metal Ions](#)

[Note: It would be helpful to draw a diagram to remember where the drops are on the sheet of metal before you begin.](#)

[ABDC](#)

[Part 4: Flame Tests](#)

[This station will be located inside a fume hood](#)

[Chapter 4. Going Green With Al](#)

[4.1.](#)

[Chapter 5. VSEPR: Molecular Shapes and Isomerism](#)

[5.1.](#)

[Grading](#)

[Chapter 6. Nanotechnology: Ferrofluids and Liquid Crystals](#)

[6.1.](#)

[Nanotechnology: Ferrofluids and Liquid Crystals](#)

[Objective](#)

[Grading](#)

[Background Information](#)

[Experimental Synthesis](#)

[Part I. Synthesis of Gold Nanoparticles \(DEMO\)](#)

[Nanoparticle Synthesis](#)

[Part II. Synthesis of the Aqueous Ferrofluid \(Procedure modified from J.](#)

[Chem. Edu. 1999, 76, 943-948.\)](#)

[Chemicals](#)

[Magnetite Synthesis](#)

[Ferrofluid Properties](#)

[Part III. Synthesis of Cholesteryl Ester Liquid Crystals](#)

[Chemicals](#)

[Liquid Crystal Synthesis](#)

[Chapter 7. Solid State and Superconductors](#)

[7.1.](#)

[Solid State Structures and Superconductors](#)

[Objectives](#)

[Grading](#)

[Before coming to lab:](#)

[Introduction](#)

[Stoichiometry](#)

[Binding forces in a crystal](#)

[Close-packing](#)

[Packing of more than one type of ion \(binary compounds\) in a crystal](#)

[lattice](#)

[Coordination number and interstitial sites](#)

[Synthesis of solid state materials](#)

[X-ray crystallography](#)

[Superconductors](#)

[Solid State Model Kits](#)

[Use of the Solid State Model Kit:](#)

[Working groups and teams](#)

[Experimental Procedure](#)

[2. Cubic Cells](#)

[Team A](#)

[Group 2. Extended Structure](#)

[4. Interstitial sites and coordination number \(CN\)](#)

[Team A](#)

[5. Ionic Compounds](#)

[Team C](#)

[Fluorite: Calcium fluoride](#)

[Team D](#)

[Lithium Nitride](#)

[· Use the L template and insert 6 rods in the parallelogram portion of the dotted lines.](#)

[· Construct the pattern shown below. Be sure to include a z=1 layer. 1 is a green sphere while 1 and 2 are blue spheres. The 0 indicates a 4.0 mm spacer tube; the 2 is an 18.6 mm spacer.](#)

[Teams E and F](#)

[Zinc Blende and Wurtzite: Zinc Sulfide](#)

[Pre-Lab: Solid State and Superconductors](#)

(Total 10 Points)

Report: Solid State and Superconductors

A. Simple Cubic Unit Cells or Primitive Cubic Unit Cells (P)

B. Body-Centered Cubic (BCC) Structure

C. The Face Centered Cubic (FCC) Unit Cell

a. Fill out the following table for a FCC unit cell.

IV.Ionic Solids

Chapter 8. Colorful Copper

8.1.

Experiment 3: Colorful Copper

Objective

Grading

Before Coming to Lab...

Introduction

Experimental Procedure

Chapter 9. Metathesis: To Exchange or Not?

9.1.

Lab 4: Metathesis: To Exchange or Not

Objectives

Grading

Before Coming to Lab...

Introduction

Formation of a Gas

Formation of a Weak Electrolyte

Table 1 Solubility Rules

[Table 2 Strong Electrolytes](#)

[Experimental Procedure](#)

[Part 1: Metathesis Reactions](#)

[Part 2: Solubility, Temperature and Crystallization](#)

[Pre-Lab 4: Metathesis – To Exchange or Not](#)

[Report 4: Metathesis – To Exchange or Not](#)

[Part 1: Metathesis Reactions](#)

[Part 2: Solubility, Temperature and Crystallization](#)

[First Batch of Crystals](#)

[Second Batch of Crystals](#)

[Third Batch of Crystals](#)

[Chapter 10. Electrochemistry/Alchemy: Molar Mass of Cu and Turning Cu into Au](#)

[10.1.](#)

[Electrochemistry/Alchemy:](#)

[Molar Mass of Cu and Turning Cu into Au](#)

[Objectives](#)

[Grading](#)

[Before Coming to Lab ...](#)

[Introduction](#)

[Background Information](#)

[Experimental Procedure](#)

[Alchemy: Copper into Gold](#)

[Example of the calculation of Molar Mass:](#)

[Electrochemistry Pre-lab Exercise](#)

[Report Form: Electrochemistry](#)

[Questions:](#)

[Alchemy - Copper into gold](#)

[Chapter 11. Bonding 07](#)

[11.1.](#)

[Lab 5: Bonding 07](#)

[Objective](#)

[Grading](#)

[Background Information](#)

[Experimental Procedure](#)

[Part I. Predicting bond type through electronegativity differences.](#)

[Part II. Weak and strong electrolytes](#)

[Chemicals](#)

[Instructions for MicroLab Conductivity Experiment](#)

[Part III. Electrolyte strength and reaction rate](#)

[Chemicals](#)

[Part IV. Chemical reactions](#)

[Chemicals](#)

[Pre-Lab 5: Bonding 07](#)

[\(Total 10 Points\)](#)

[Part I. Bonding of chemicals in solution](#)

[Report 5: Bonding 07](#)

[Part I. Predicting bond type through electronegativity differences.](#)

[Part II. Weak and strong electrolytes](#)

[Part III. Electrolyte strength and reaction rate](#)

[Part IV. Chemical reactions](#)

Chapter 12. Silver Nanoparticles: A Case Study in Cutting Edge Research

12.1.

Objective

Background

What is nanotechnology?

Why care about nanotechnology?

What are nanoparticles and how are they made?

Why silver nanoparticles?

TA Demonstration on the Hydrophobicity of Silver

Experimental Procedure no1 - ripening of silver nanoparticle

Experimental procedure no2 - lasers and colloids

Additional Information

Chapter 13. Thermochemistry

13.1.

Thermochemistry

Objective

Grading

Background Information

Calorimetry

Experimental Procedure

Materials Required

Setup of the MicroLab Thermistor

Part I. Reaction of Strong Acid with Strong Base

Part II. Enthalpy of Reaction

Calculations

Chapter 1. Avogadro and All That

Experiment 2: Avogadro and All That

Objective

To help you become familiar with the layout of the laboratory including safety aids and the equipment that you will be using this year.

To make an order-of-magnitude estimate of the size of a carbon atom and of the number of atoms in a mole of carbon, based on simple assumptions about the spreading of a thin film of stearic acid on a water surface

Grading

Lab Report (90%)

TA points (10%)

Before coming to Lab

Read the following:

Lab instructions

Background Information

Concepts of the experiment

Print out the lab instructions and report form.

Read and sign the equipment responsibility form and the safety rules. Email Ms. Duval, at nduval@rice.edu, to confirm completing this requirement by noon on September 14 th.

Introduction

Since chemistry is an empirical (experimental) quantitative science, most of the experiments you will do involve measurement. Over the two semesters, you will measure many different types of quantities – temperature, pH, absorbance, etc. – but the most common quantity you will measure

will be the *amount* of a substance. The amount may be measured by (1) weight or mass (grams), (2) volume (milliliters or liters), or (3) determining the number of moles. In this experiment we will review the methods of measuring mass and volume and the calculations whereby number of

moles are determined.

Experimental Procedure

1. Identification of Apparatus

On the tray (in DBH 214) we have a number of different pieces of common equipment. We will, identify and sketch each - I know this may sound a trivial exercise but it is necessary so that we are all on the same page.

1. beaker
2. Erlenmeyer flask (conical flask)
3. side-arm Erlenmeyer flask
4. graduated (measuring) cylinder
5. pipettes, both types graduated and bulb
6. burette
7. Bunsen burner
8. test tubes
9. watch glass
10. volumetric flask

2. Balance Use

In these general chemistry laboratories, we only use electronic balances – saving you a lot of time.

However, it is important that you become adept at using them.

Three aspects of a balance are important:

1. The on/off switch. This is either on the front of the balance or on the back.
2. The "Zero" or "Tare" button. This resets the reading to zero.
3. **CLEANLINESS.** Before and after using a balance, ensure that the entire assembly is spotless.

Dirt on the weighing pan can cause erroneous measurements, and chemicals inside the

machine can damage it.

Balance Measurements:

1. Turn the balance on.
2. After the display reads zero, place a piece of weighing paper on the pan.
3. Read and record the mass. (2)
4. With a spatula, weigh approximately 0.2 g of a solid, common salt NaCl. The excess salt is discarded, since returning it may contaminate the rest of the salt.
5. Record the mass (1). To determine how much solid you actually have, simply subtract the mass of the weighing paper (2) from the mass of the weighing paper and solid (1). Record this mass (3). You have just determined the mass of an "unknown amount of solid."
6. Now place another piece of weighing paper on the balance and press the Zero or Tare button then weigh out approximately 0.2 g of the salt (4). Thus, the zero/tare button eliminates the need for subtraction.

3. Measuring the volume of liquids

When working with liquids, we usually describe the quantity of the liquid in terms of volume, with the usual units being milliliters (mL). We use three types of glassware to measure volume –

(1) burette, (2) bulb pipette, and (3) graduated cylinder. A volumetric flask will also allow for a high degree of accuracy and precision in the measurements of any liquids, so a 100 mL volumetric flask will contain precisely 100.0 mL of solution when filled to the line marked on the neck of the flask.

Examine each piece of equipment. Note that the sides of each are graduated for the graduated cylinder and the burette. The bulb pipette delivers a specific volume, 10.00 mL. The burette will be used to deliver a variable volume of solution and will also be precise to two decimal places.

Put some water into the graduated cylinder. Bend down and examine the side of the water level.

Note that it has a "curved shape." This is due to the water clinging to the glass sides and is called the meniscus. When reading any liquid level, use the center of the meniscus as your reference point.

Graduated cylinder

Look at the graduations on the side of the cylinder. Note that they go from 0 on the bottom and increase upwards. Since volumes in graduated cylinders are only precise to one decimal place, *a graduated cylinder is generally only used when a high degree of precision is not required.*

1. Using your 10mL graduated cylinder, add water up to the 10 mL line as accurately as possible.
2. Dry a small beaker and weigh it (2).
3. Pour the 10 mL of water from the cylinder into the beaker. Reweigh (1).
3. Pour the 10 mL of water from the cylinder into the beaker. Reweigh (1).
4. Subtract the appropriate values to get the weight of the water (3).

Bulb Pipette

1. Half-fill a beaker with water.
2. Squeeze the pipette bulb and attach to the top of the pipette. Put the spout of the pipette under water and release the bulb. It should expand, drawing the water into the pipette. **Do not let the water be drawn into the bulb.**
3. When the bottom of the meniscus is above the line on the pipette, remove the pipette from the water.
4. Squeeze the bulb to run the extra water into a waste container until the bottom of the meniscus is level with the line on the pipette.
5. Add 10 mL of water to a pre-weighed dry beaker (5).
6. Weigh (4).
7. Subtract to get the weight of the water (6).

Burette

1. Examine the graduations. Note that 0 is at the top. Note that the stopcock is horizontal to close the burette and vertical to open it.
2. First, lower the burette so that the top is easy to reach and make sure the burette is closed.

Using a funnel, add about 10 mL of water.

3. Open the burette and run a little water into a waste container. Then turn the burette upside down and allow the rest of the water to run into the container.
4. You have just rinsed your burette. This should be done every time before using a burette – first rinse with water, then repeat the process using whatever liquid is needed in the experiment.

5. Fill the burette to any convenient level (half-way is fine). It is a good technique to add more liquid than you need, and allow some liquid to run into a waste container until you reach the appropriate level so that you fill the space from the top to the tip of the burette.
6. Dry a beaker and weigh (8).
7. Add 10 mL of water to a pre-weighed dry beaker (7).
8. Subtract to get the weight of the water (9).

4. Estimation of Avogadro's number

Briefly, as a group with your TA, you will make an approximate (order of magnitude) estimate of Avogadro's number by determining the amount of stearic acid that it takes to form a single layer (called a monolayer) on the surface of water. By making simple assumptions about the way the stearic acid molecules pack together to form the monolayer, we can determine its thickness, and from that thickness we can estimate the size of a carbon atom. Knowing the size of a carbon atom, we can compute its volume; and if we know the volume occupied by a mole of carbon (in the form of a diamond), we can divide the volume of a mole of carbon by the volume of an atom of carbon to get an estimate of Avogadro's number.

Procedure

Special Supplies: 14 cm watch glass; cm ruler; polyethylene transfer pipets; 1-mL syringes; pure distilled water free of surface active materials; disposable rubber gloves (for cleaning own watch glasses in 0.1 M NaOH in 50:50 methanol/water): 13 100 mm test tubes with rubber stoppers to fit.

Chemicals: pure hexane, 0.108 g/L stearic acid (purified grade) solution in hexane. 0.1 M NaOH in 50:50 methanol/water used for washing the watch glasses, dye.

SAFETY PRECAUTIONS: Hexane is flammable! There must be no open flames in the laboratory while hexane is being used.

WASTE COLLECTION: At the end of the experiment, unused hexane solvent and stearic acid in hexane solution should be placed in a waste container, marked "Waste hexane/stearic acid solution in hexane."

Measurement of the volume of stearic acid solution required to cover the water surface

Your TA will do this as a group demonstration:

1. Fill the clean watch glass to brim with deionized water. One recommended way to do this is to set up your 25 mL burette on a ring stand.
2. Using a transfer pipette, obtain about 3-4 mL 0.108 g/L stearic acid solution in hexane in a clean, dry 13 100 mm test tube. Keep the tube corked when not in use.
3. Obtain more distilled water and fill the burette. Place your watch glass directly under the burette (about 1 inch or less from the tip) and dispense the water until the entire watch glass is full. You may have to refill the burette 4 or 5 times to do this. With careful dispensing, the surface tension of the water should allow you to fill the entire watch glass with relative ease.
4. Carefully measure the diameter of the water surface with a centimeter ruler. It should be close to 14 cm, + or - a couple of millimeters. Next, rinse and fill your 1 mL syringe with stearic acid solution, taking care to eliminate bubbles in the solution inside the syringe.
5. Read and record the initial volume of the syringe (1 mL is always a good place to start.)
6. Then add the stearic acid solution drop by drop to the water surface. Initially, the solution will spread across the entire surface, and it will continue to do so until a complete monolayer of stearic acid has been formed. If your first few drops do not spread and evaporate quickly, either your water or watch glass is still dirty. As this point is approached, the spreading will become slower and slower, until finally a drop will not spread out but will instead sit on the surface of the water (looking like a little contact lens). If this "lens" persists for at least 30 s, you can safely conclude that you have added 1 drop more than is required to form a complete monolayer.

7. Now, read and record the final volume reading of the syringe.

When you have completed all of your measurements, rinse your syringe with pure hexane, and dispose of all the hexane-containing solutions in the waste collection bottle provided.

Calculation of Avogadro's Number

The calculation proceeds in several steps.

We calculate the volume of stearic acid solution in hexane required to deliver enough stearic acid to form a monolayer.

All of the hexane evaporates, leaving only the thin monolayer film of stearic acid, so we next calculate the actual mass of pure stearic acid in the monolayer.

We calculate the thickness of the stearic acid monolayer, using the known density of stearic acid and the area of the monolayer.

Assuming the stearic acid molecules are stacked on end and are tightly packed, and knowing that there are 18 carbon atoms linked together in the stearic acid molecule, calculate the diameter and volume of a carbon atom.

Calculate the volume of a mole of carbon atoms in diamond; divide the molar volume of carbon (diamond) by the volume of a single carbon atom to obtain an estimate of Avogadro's number.

Remember that the units of Avogadro's number are mol^{-1} , so you can use unit analysis to check your answer.

Solutions

Chapter 2. Stoichiometry: Laws to Moles to Molarity

Experiment 3: Stoichiometry: Laws to Moles to Molarity

Objective

To determine the mass of a product of a chemical reaction

To make a solution of assigned molarity

To test the solubility of Peeps, yes Peeps, in various solvents

Grading

Lab Report (90%)

Quiz(10%)

Before Coming to Lab...

Read the lab instructions

Complete the online quiz, this is part of your grade NOT bonus

Introduction

The word stoichiometry derives from two Greek words: *stoicheion* (meaning "element") and *metron* (meaning "measure"). Stoichiometry deals with calculations about the masses (sometimes volumes) of reactants and products involved in a chemical reaction. Consequently, it is a very

mathematical part of chemistry.

In the first part of this lab, sodium bicarbonate is reacted with an excess of hydrochloric acid.

(2.1)



By measuring the mass of NaHCO₃ and balancing the equation (above), the mass of NaCl expected to be produced can be calculated and then checked experimentally. Then, the actual amount of NaCl produced can be compared to the predicted amount.

This process includes molar ratios, molar masses, balancing and interpreting equations, and

$$C_M = \frac{n_{\text{substance}}}{V_{\text{solution}}}$$

conversions between grams and moles and can be summarized as follows:

1. Check that the chemical equation is correctly balanced.
2. Using the molar mass of the given substance, convert the mass given in the problem to moles.
3. Construct a molar proportion (two molar ratios set equal to each other). Use it to convert to moles of the unknown.
4. Using the molar mass of the unknown substance, convert the moles just calculated to mass.

In the second part of this lab, since a great deal of chemistry is done with solutions, a solution will be prepared of allocated molarity. Molarity, or more correctly molar concentration, is defined to be the number of moles of solute divided by the number of liters of solution:

Figure 2.1.

with units of [mole/L]. However molar concentration depends on the temperature so a higher temperature would result in an increased volume with a consequential decrease in molar concentration. This can be a significant source of error, of the same order as the error in the volume measurements of a burette, when the temperature increases more than 5 °C.

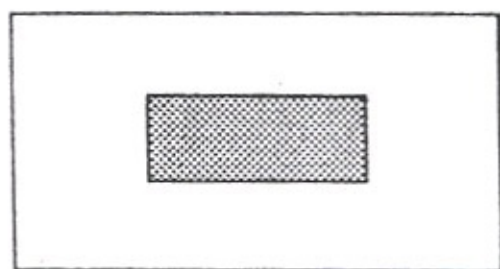
Steps to preparing a solution of a certain concentration:

1. First, you need to know the formula for the solute.

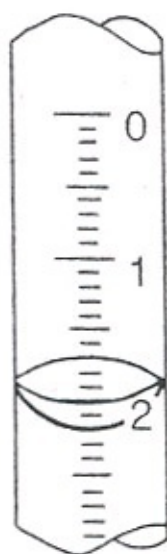
2. Next, you need to calculate the molecular weight of the solute by adding up the atomic weights of the elements present in the correct ratios.
3. Then, based on the volume of solution you are making, calculate the mass of solute needed to dissolve in the solution volume. Usually, deionised water is the solvent.
4. Remember to ensure that all the solute is dissolved before finally filling to the mark on the volumetric flask. If there is any undissolved solute present in the solution, the water level will go down slightly below the mark, since the volume occupied by the solute differs from the actual volume it contributes to the solution once it is dissolved.

Example solution preparation: potassium chromate

1. The formula for potassium chromate is K_2CrO_4 .
2. The elements present are potassium, chromium, and oxygen with atomic masses of 39.10,



buret reader



volume = 2.00 mL

2. The elements present are potassium, chromium, and oxygen with atomic masses of 39.10, 52.00 and 16.00 respectively. Adding up these numbers in the correct ratios dictated by the formula yields the following: $2 \times 39.10 + 1 \times 52.00 + 4 \times 16.00 = 194.20$ g/mol.

3. For one liter of solution use a 1000 mL volumetric flask. So a 1M solution would require

194.2g of solid K_2CrO_4 in 1 L, 0.1M 19.42g of solid K_2CrO_4 and so on.

Your teaching assistant will check the accuracy of the solution that you have made by titration, which is a method of quantitatively determining the concentration of a solution. A

standardsolution (a solution of known concentration) is slowly added from a burette to a

solution of the analyte (a solution of unknown concentration – your solution) until the reaction

between them is judged to be complete (equivalence point). In colorimetric titration, some

indicator must be used to locate the equivalence point. One example is the addition of acid to

base using phenolphthalein (indicator) to turn a pink solution colorless in order to determine the

concentration of unknown acids and bases. Record your TA's value of the molarity of your solution on your report form along with your percent error.

Figure 2.2.

Figure 1: Reading the Burette

When an acid is neutralized by a base, there are stoichiometrically equal amounts of acid and base and the $pH = 7$. It is possible to accurately determine the concentration of either the acid or base solution.

Moles of a substance = Concentration of solution (moles/L) x Volume (L)

We can calculate the concentration of the acid or base in the solution by using the following

equation where balance base and balance acid refer to the stoichiometric ratio of the base and acid to each other.

(2.2)

Balance Base (B_b) x Moles of Acid = Moles of Base x Balance Acid (B_a)

(2.3)

$B_b \times C_a \times V_a = B_a \times C_b \times V_b$ **Titration Calculations:**

Step 1: Balance the neutralization equation. Determine Balance of Acid and Base.

Step 2: Determine what information is given.

Step 3: Determine what information is required.

Step 4: Solve using the equation below.

$B b \times C a \times V a = B a \times C b \times V b$ **Example:**

Calculate the concentration of a nitric acid solution HNO_3 if a 20 mL sample of the acid required an average volume of 55 mL of a 0.047 mol/L solution of $\text{Ba}(\text{OH})_2$ to reach the endpoint of the titration.

Step 1: $2\text{HNO}_3 + \text{Ba}(\text{OH})_2 \rightarrow \text{Ba}(\text{NO}_3)_2 + 2\text{H}_2\text{O}$ Balance Base = 1 Balance Acid = 2

Step 2: Given information Volume Acid = 20 mL Volume Base (average) = 55 mL Concentration of Base = 0.047 mol/L

Step 3: Required information Concentration of Acid **Step 4:** Solve using the equation.

$B b \times C a \times V a = B a \times C b \times V b$ $1 \times C a \times 20 \text{ mL} = 2 \times 0.047 \text{ mol/L} \times 55 \text{ mL}$ $C a = 0.2585 \text{ mol/L}$ (considering significant figures 0.26 mol/L)

Procedure

Materials List

sodium bicarbonate NaHCO_3

3M hydrochloric acid (HCl) solution

Part 1

1. Weigh an empty 150-mL beaker on the electronic balance. Record this value in your data table.

2. Remove the beaker from the balance and add one spoonful of sodium bicarbonate. Re-weigh and record this value. Use a small spoonful.

3. Subtract the mass of the beaker and sodium bicarbonate from the mass of the beaker to get the mass of the sodium bicarbonate added.

4. Pour approximately 20 mL of 3M hydrochloric acid into a 100-mL beaker. Rest a Pasteur pipette in the beaker.

5. Add 3 drops of acid to the NaHCO_3 beaker, moving the pipette so that no drops land on each other. The key point is to spread out the addition of acid so as to hold all splatter within the walls of the beaker.

6. Continue to add acid slowly drop by drop. As liquid begins to build up, gently swirl the beaker.

This is done to make sure any unreacted acid reaches any unreacted sodium bicarbonate. Do not add acid while swirling.

7. Stop adding the hydrochloric acid when all bubbling has ceased so that the minimum amount of HCl has reacted with all of the sodium bicarbonate. Check when all the bubbling has ceased, by swirling the beaker and to ensure that there is no more bubbling. When all the bubbling has ceased add one drop more of acid and swirl.

8. Using a microwave oven, dry to constant weight. Initially place the beaker in the microwave for 1 min when there is plenty of solution present. Then in 5 second intervals thereafter.

Drying to a constant weight means that when you think your sample is dry, you weigh the beaker, dry for 5 more seconds, weigh the beaker again, dry for 5 more seconds, and weigh the beaker one last time. The three masses should be within one milligram. If they are not, continue to dry your sample until they are.

9. Calculate the percent yield of NaCl. Percent yield can be calculated as follows:

(2.4)

Materials List

100 mL volumetric flask

3M hydrochloric acid (HCl) solution

sodium bicarbonate (NaCHO₃)

methyl red indicator

Part 2

1. Ask you TA for your assigned molarity – it will range from 0.1M to 1M.

2. From the formula of the solute, find the molecular weight of the solute, in g/mol.

3. Determine how many grams of sodium bicarbonate you will need to make a 100mL solution

for the assigned molarity.

- Using your 100mL volumetric flask, add the correct amount of sodium bicarbonate. Make sure to use a funnel to pour the solute into the flask. Rinse the funnel with deionised water to make sure all of the solute makes it into the volumetric flask. Fill the volumetric flask half way with water, cap it, and invert repeatedly until all of the solute is dissolved. Hold your thumb over the cap to make sure that the flask does not leak. Once all of the solute is dissolved, remove the cap, fill the flask to the mark with water, recap the flask, and invert several more times.
- Take your solution to your TA to check the molarity by titration, record value on your report form and your percent error. Percent error can be calculated as follows:

(2.5)

Solutions

Chapter 3. Totally, Terrific Table

Lab 4: Totally, Terrific Table!

Objective

The goals of this experiment are:

To observe the reactions of several metals with cold water, hot water, acids, and other metal ions.

To prepare an activity series of the metals based on the observations from the above reactions.

Grading

You will be assessed on:

Observations of the reactions of several metals with cold water, hot water, acids and then other metal ions.

Preparation of an activity series of the metals based on the observations from the above reactions.

Answers to the quiz and post-lab questions.

Background Information

First, you are going to travel back to 1869 and marvel at how the first periodic law and table were born when only 63 elements had been discovered at the time. A 35 year old professor of general

chemistry, **Dmitri Ivanovich Mendeleev**, at the University of St. Petersburg (now Lennigrad) in Russia was shuffling cards, each labeled with the property of an element, trying to organize his thoughts for his soon-to-be famous textbook on chemistry. He realized that if the elements were

arranged in the order of their atomic weights, there was a trend in properties that repeated itself several times!

In order to see and find order among the elements, we must have some general acquaintance with them. Elements are made of matter, and matter is defined as anything that has mass and occupies space. This includes everything that you can see and a lot that you cannot. It follows that in order to distinguish between different types of matter (in other words different elements) we have to assess their properties.

There are two types of properties: intensive and extensive. In the former case, **intensive** properties do not depend on the how much of an element is present but do include state (whether a substance is a solid, liquid or gas), color and chemical reactivity. **Extensive** properties depend on the quantity of matter present; mass and volume are extensive properties.

Properties can be further categorized as either chemical or physical. A chemical change describes how the substance may change composition, such as spontaneously by combustion or in

combination with other substances. On the other hand, physical changes are those properties that can be measured without changing the composition of the matter. Condensation of steam to water

is a physical change.

Introduction

What is there to know about the periodic table? Why is it important? Why does it appear in nearly every science lecture room and lab? Is it just a portrait of an aspect of chemistry or does it serve a useful purpose? Why is the name **periodic** appropriate? Why is the table arranged in such a way?

What are the important features of the table? Does it give order to the approximately 120 known elements?

Relative Reactivity of Metals and the Activity Series

A superficial glance at the Periodic Table will reveal that all known elements are listed by their chemical symbols. An in depth glance at the Periodic Table yields information on the mass of an

atom of the element in atomic mass units (amu) for the molar mass of a mole (6.02×10^{23}) of

atoms in grams below the chemical symbol for each element. Above the chemical symbol for each

element, there is a second number listed, the atomic number, which gives the number of protons

(positively charged particles in the nucleus), or the number of electrons (negatively charged outside the nucleus) for a neutral atom.

Mendeleev arranged the elements in the Periodic Table in order of increasing atomic number in horizontal rows so that elements with similar properties recur periodically (another words they fall directly beneath each other in the Table). The elements in a given vertical column are referred to as a family or group. The physical and chemical properties of the elements in a given family change gradually as one goes from one element in the column to the next. By observing the trends in properties, the elements can be arranged in the order in which they appear in the Periodic Table.

Procedure

I. Activity Series

Part 1: Reactions of Metals with Water

CAUTION: Sodium and potassium react very rapidly with water to evolve hydrogen and heat. This is potentially dangerous because of the possibility of the violent explosive reaction of $H_2(g)$ with $O_2(g)$ present in the air. Both sodium and potassium cause severe chemical burns when it comes into contact with the skin.

CAUTION: H_2 is flammable.

Demo: The reactions of sodium and potassium with water will be demonstrated for you. Observe the rate of evolution of H_2 gas as a tiny piece of sodium or potassium is placed in a 500-mL beaker full of deionized water. Record your observations on your report form and write a balanced equation for these reactions.

1. Place 5 mL H_2O (cold water should be used for Cu, Zn and Ca and warm water should be used with Mg) in each of four clean test tubes and place them in a test tube rack. Label them as follows:

Table

3.1.

A. Mg

B. Cu

C. Zn

D. Ca

1. Place several small pieces of Mg, Cu, and Zn in the correctly labeled test tube prepared above.

Place **two or three** pieces of Ca turnings in the test tube labeled "Ca".

2. Watch for evidence of reaction by noting evolution of gas bubbles and any change in the color or size of the metal. Be patient, some reactions may be slow! Record your observations and

write net ionic equations for each reaction.

3. **Discard the Ca and water in the Ca recovery beaker.** Decant the water out of the test tubes containing Mg, Cu, and Zn and leave the pieces of metal that remain unreacted in each test

tube.

Note: Trapped air bubbles on the metal surfaces are not indicative of a reaction.

Note: Net ionic equations must balance in mass (atoms) and in total charge on each side of the equation.

Part 2: Reactions of Metals with HCl

CAUTION: This part of the procedure must be done in a fume hood!!

CAUTION: Some of the test tubes may become very hot. Leave them in the test-tube rack while you are making observations.

1. Place the **three** remaining test tubes in a test tube rack. There should be only **three** test tubes remaining.

2. Add 2 mL of 3 M HCl solution to each test tube.

3. Observe the relative rate of H₂ gas evolution for up to 5 minutes and record your observations on your report form.

4. Based on the observations in the previous steps, list the elements that react in 3M HCl in order of increasing strength as reducing agents and write net ionic equations for all reactions.

Part 3: Reactions of Metals with Other Metal Ions

Note: It would be helpful to draw a diagram to remember where the drops are on the sheet of metal before you begin.

ABDC

1. Place a clean 1 inch-square of metal foil (sheet) of each of these metals Cu, Zn and Pb on a flat

surface.

2. Clean one side of the metal surface by sanding it with fine sandpaper or steel wool.

3. Place one or two drops in spots of each of these solutions in a clockwise order on the metal surfaces:

Table 3.2.

A. 0.5 M Ag^+

B. 0.5 M Cu^{2+}

C. 0.5 M Zn^{2+}

D. 0.5 M Pb^{2+}

NOTE: Do not test a metal with a solution of the same metal ion, such as Cu metal with Cu^{2+} ion.

1. Watch for color changes in each spot as evidence of reaction. If you are not sure whether the reaction has occurred, rinse the plate with water. A distinct spot of a different color on the surface is good evidence for the reaction.

2. Write net ionic equations for each reaction. Arrange Ag, Cu, Pb and Zn in order of their

increasing strength as reducing agents. If a metal **A** reacts with a cation of another metal **B**, metal **A** is a stronger reducing agent, more reactive than metal **B**.

3. Rinse and dry each square of metal and return it to the correct beaker on the reagent shelf for other students to use.

Part 4: Flame Tests

This station will be located inside a fume hood

Chemicals/Materials:

0.5 M Solutions of dissolved chloride salts of: Li, Na, K, Rb, Cs, Ca, Ba, Cu, Pb, Fe(II), and Fe(III), Sr (nitrate salt)

Wooden splints, one per each solution

Bunsen burner/clicker

1M HCl

For each solution, take the splint out of the beaker and pass it through the flame of the Bunsen burner,

remembering that the hottest part of the flame is the blue section in the center. Do not leave the splint in the flame too long as it will eventually catch on fire.

Replace the splint in the same beaker from which it came.

On your report form, record the color of the flame of each metal.

Solutions

Chapter 4. Going Green With Al

Going Green With Al

Objectives

To prepare common alum, $KAl(SO_4)_2 \cdot 12H_2O$, from a discarded aluminum beverage can.

To perform qualitative analysis on alum.

To investigate the acid-base behavior of aluminum compounds.

Grading

Your grade will be determined based on the following:

Quiz (10%)

Lab Report Form (90%)

Before coming to lab...

Read the lab instructions

Print out the lab instructions and report form, take the online quiz

Introduction

Aluminum is the most abundant metal in the earth's surface (7.5% by mass). Rubies and sapphires contain aluminum and it is used in a range of products from rocket fuel to antiperspirants. All of these products contain aluminum metal or aluminum compounds of various kinds. The abundance of aluminum, coupled with its attractive combination of physical and chemical properties, accounts for the fact that it is one of the principal industrial raw materials used by industrialized societies. Production of aluminum from raw materials is an energy intensive process.

Since the metal is not consumed rapidly by corrosion, the amount of scrap aluminum grows rapidly while the available supply of raw materials for the manufacture of aluminum decreases.

The average predicted longevity of an aluminum can along the roadside is 100 years.

Environmental problems thus created are typical of those of several different metals. One obvious solution to the problem is to recycle the used fabricated aluminum into other useful metallic objects or into aluminum compounds. Aluminum metal can be recovered from scrap by melting the metal and separating it from solids and volatile impurities. This process uses a large amount of energy. The energy requirement to prepare an aluminum can from recycling is only 5% of the energy required to produce the can from bauxite ore.

This experiment illustrates a chemical recovery process in which waste aluminum is converted chemically into an aluminum compound, hydrated potassium aluminum sulfate, $KAl(SO_4)_2 \cdot 12 H_2O$, or common alum. Although alum is an important industrial compound, the method of preparation in this experiment is not the way alum is obtained for use in industry. Nevertheless, this experiment will illustrate an interesting example of the reduction of environmental waste.

"Alum" is a generic term that describes hydrated double salts of certain metals having the generalized formula, $[MM' (SO$

+

$4)2.12 H_2O]$, in which M (univalent) is commonly Na^+ , K^+ , NH_4^+ ,

or Rb^+ and M' (trivalent) is commonly Al^{3+} , Ga^{3+} , V^{3+} , Cr^{3+} , Mn^{3+} , Fe^{3+} , or Co^{3+} . True alums

crystallize in well-defined octahedral shapes and many are beautifully colored, particularly those containing *d*-block transition metals. The ancient Egyptians, Greeks and Romans used alum as a mordant in dyeing cloth, which we will meet again in our last lab for this semester. A mordant

contains metal ions that bind dyes to the fabric. Presently alum is used to harden photographic film, to prepare pickles, as a mordant, and for other purposes.

Experimental Procedure

Wear safety goggles at all times during this lab.

Wear gloves when using concentrated acid and base.

For this experiment there are two samples of aluminum that will be used. Some of the steps will

be the same for both cans, but some will be different so it is essential that you keep track of the samples. The best way to ensure there is no confusion is for Group A to do the experiment with

sample #1 and Group B to do the experiment with sample #2. However, it is still important to

understand what your other group is doing so try to be as involved as possible as you are carrying out your part.

Sample #1 (Group A)

Part 1: Preparation of Alum

1. Cut a 5cm x 5cm square piece of aluminum from a scrap aluminum can and sand the paint off using steel wool.
2. Cut this piece into smaller pieces (about 0.5 cm long) and weigh out ~0.5 g. Record the weight to 3 decimal places.
3. Place the aluminum in a 400-mL beaker and add 25 mL of 4 M KOH.
4. Place beaker on the heating mantle and heat gently.

CAUTION - H₂ GAS (VERY EXPLOSIVE) IS PRODUCED. ENSURE THAT YOU ONLY HEAT THE BOTTOM OF THE BEAKER

1. When the bubbles have stopped, remove from the heat.
2. Vacuum filter the solution. This is done by taking a filter funnel, placing a piece of wet filter paper in it, placing the funnel in a vacuum flask, and attaching the flask to the vacuum line, labeled "vac" on the outlet taps. Save the filtrate (the liquid inside the filter flask). You may dispose of the solid in the marked waste beaker.
3. Clean the 400 mL beaker you used before and pour the filtrate into it.
4. Carefully, rinse your 100 mL graduated cylinder. Dispense 15 mL of 3 M H₂SO₄ into it, which you will then add to the filtrate.

CAUTION - H₂SO₄ IS A STRONG ACID AND DEHYDRATOR. SEE TA IMMEDIATELY IF YOU SPILL ANY!

USE EXTREME CAUTION WHEN CLEANING YOUR GRADUATED CYLINDER AFTER IT HAS COME INTO CONTACT WITH THE SULFURIC ACID. BE SURE TO WEAR GLOVES WHILE HANDLING IT AT ALL TIMES!!

1. A white powder of Al(OH)₃ should form.
2. Vacuum filter this solution, using a clean piece of weighing paper, and save the solid Al(OH)₃.

Part 2: Qualitative Analysis of Alum

1. Use a spatula to transfer a few of the $\text{Al}(\text{OH})_3$ crystals (about 5 mg) to a watch glass. Add 3 drops of water to the crystals. Stir gently until the crystals dissolve.
2. Use a small piece of indicator paper to see whether the solution is acidic, basic, or neutral.
3. Now add 1 drop of 0.5 M BaCl_2 (barium chloride) to the solution. Record your observations.
4. As you now know, a really good test for the presence of potassium is a flame test. Using the hot grips, hold a spatula in the flame of a Bunsen burner to volatilize impurities from the spatula.
5. When one end of the spatula is red hot, remove it, and quickly touch it to a small cluster of crystals. Several should stick.
6. Slowly bring the spatula (plus crystals) toward the flame and watch carefully. Hold the crystals in the flame for at least 5 seconds (until the solid glows). Record your observations.
7. Dispose of the $\text{Al}(\text{OH})_3$ and related compounds in the marked waste beaker.

Sample #2 (Group B)

Part 1: Preparation of Alum

1. Cut a 5cm x 5cm square piece of aluminum from a scrap aluminum can and sand the paint off using steel wool.
2. Cut this piece into smaller pieces (about 0.5 cm long) and weigh out ~0.5 g. Record the weight to 3 decimal places.
3. Place the aluminum in a 400-mL beaker and add 25 mL of 4 M KOH.
4. Place beaker on the heating mantle and heat gently.

CAUTION - H_2 GAS (VERY EXPLOSIVE) IS PRODUCED. ENSURE THAT YOU ONLY HEAT THE BOTTOM OF THE BEAKER

1. When the bubbles have stopped, all of the aluminum will have reacted. Remove from the heat temporarily

Part 2: Calculation of Percent Yield

1. Vacuum filter the solution. This is done by taking a filter funnel, placing a piece of wet filter paper in

it, placing the funnel in a vacuum flask, and attaching the flask to the vacuum line,

labeled "vac" on the outlet taps . Save the filtrate (the liquid inside the filter flask). You may dispose of the solid in the marked waste beaker.

2. Clean the 400 mL beaker you used before and pour the filtrate into it.

3. Carefully, rinse your 100 mL graduated cylinder. Dispense 15 mL of 3 M H₂SO₄ into it, which you will then add to the filtrate.

CAUTION - H₂SO₄ IS A STRONG ACID AND DEHYDRATOR. SEE TA

IMMEDIATELY IF YOU SPILL ANY!

USE EXTREME CAUTION WHEN CLEANING YOUR GRADUATED CYLINDER AFTER

IT HAS COME INTO CONTACT WITH THE SULFURIC ACID. BE SURE TO WEAR

GLOVES WHILE HANDLING IT AT ALL TIMES!!

1. Heat gently while stirring until the solution becomes clear. Add 2 or 3 boiling chips, and boil the solution down to a volume of 20 mL. Do not boil the solution to dryness.

2. After "boiling off", let the beaker cool to room temperature. Crystals of alum should form.

2. After "boiling off", let the beaker cool to room temperature. Crystals of alum should form.

Allow to cool for 15 minutes, or until crystals are clearly visible. Disregard any tiny black specks.

3. Once you see that the crystals are clearly formed, vacuum filter the product and wash the beaker and then the product in the funnel with a small amount of ethanol to remove any remaining crystals from the beaker and remove any impurities.

4. Transfer the remaining solid to a watch glass, scraping the filter paper with a spatula to remove as much of the product as possible.

5. Place the watch glass in the drying oven at 40 or 50 degrees (your TA should adjust this) for 10 to 15 minutes, until the white solid appears dry.

6. Remove the boiling chips and weigh.

7. Using the initial mass of aluminum and the mass of your solid, calculate the percent yield and describe the appearance of the crystals.

8. Dispose of the alum in the marked waste beaker.

Both Groups A & B

Part 3: Acid-Base Properties of Aluminum Compounds

1. Clean two test-tubes and label them 1 and 2. Place 10 drops of 1M aluminum nitrate solution in each tube. Add 10 drops of 1.0 M sodium hydroxide solution to each tube, mix well, and record your observations on your lab report.
2. Add 3 mL of 6M sodium hydroxide solution to tube 1. Mix well and record your observations.
3. Add 3 mL of 6M HCl solution to tube 2. Mix well and record your observations.
4. To tube 1, add aluminum hydroxide (the product from step 9 of Group A's prep of alum), 3 mL of 6M ammonium hydroxide, *i.e.* aqueous ammonia and record your observations.
5. Dispose of solutions in the waste beaker in the hood.

Solutions

Chapter 5. VSEPR: Molecular Shapes and Isomerism

Molecular Shapes & Isomerism

Objectives

Understand the 3-dimensional nature of molecules

Learn about Molecular Symmetry

Be able to identify the various isomers possible for one molecular formula

Be able to identify enantiomers

Grading

Quiz (10%).

Lab Report Form (90%).

Before Coming to Lab . . .

Look over the following to make sure you have a basic understanding of the topics presented.

Drawing Lewis Structures

Determining the Shapes of Molecules from their Lewis Structures

Some Basic Aspects of Bonding

Model Kits

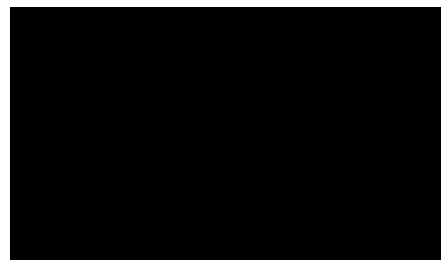
Introduction

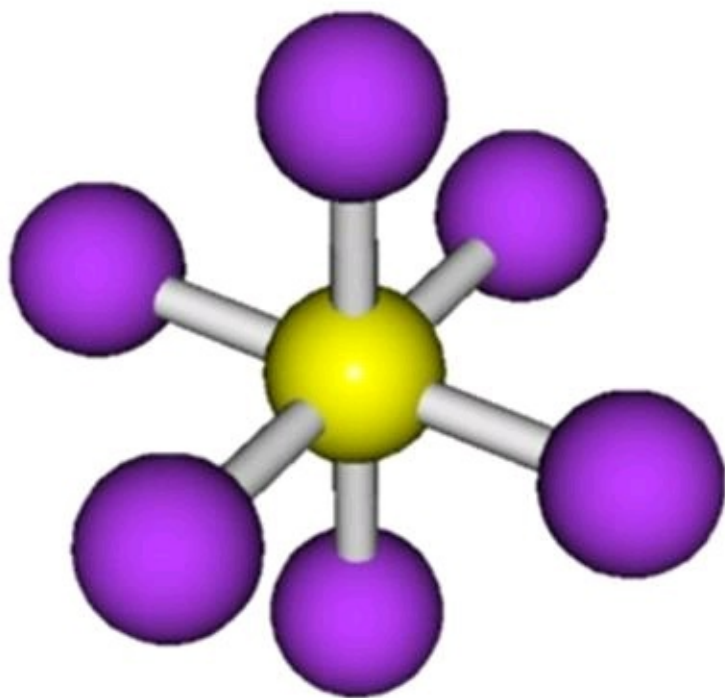
The shape of a molecule is extremely important in determining its physical properties and reactivity. A multitude of shapes are possible, and in today's lab, you will be looking at several.

In Part 1, you will be exploring the various symmetry elements that can be present in molecules.

The symmetry elements you will be looking for are mirror planes, rotation axes, and inversion centers. Being able to determine which symmetry elements are present in a molecule help in

understanding its chemistry. If there is a plane present in the molecule that has the exact same arrangement of atoms on either side of the plane, then the molecule has a **mirror plane (σ)**. It is important to note that a molecule can have more than one mirror plane. **Rotation axes** are





represented as **C_n** ($n = 1, 2, 3 \dots$). The subscript indicates how many degrees of rotation ($360^\circ/n$) are needed in order to return to the same orientation of atoms with which you started. So if there is a **C₂** axis, the rotation would be 180° . An example of a molecule having a **C₂** axis is **H₂O**.

Figure 5.1.

The third symmetry element is an **inversion center (i)**. In such molecules, starting at any position and drawing a line through the center and an equal distance to the opposite side of the molecule, you will end up at a position with an identical environment to the one you started from.

Figure 5.2.

Part 2 of the lab introduces the concept of **enantiomers**. Enantiomers are molecules sharing the same molecular configuration, but they are non-superimposable images of each other. This

concept should become clearer as you build the models for this part of the lab. Enantiomers share many of the same physical properties. The property which distinguishes them is the direction in

which they rotate plane-polarized light. They will rotate the light in equal amounts but in different directions (*plane-polarized light is just light in which all wave vibrations have been filtered out except for those in one plane*). If both enantiomers are present in a 1:1 ratio, the effects of the rotation of light cancel and no net rotation is observed. Such a mixture of isomers is known as a **racemic mixture** or as a **racemate**. Because these isomers rotate plane-polarized light, they are also known as **optical isomers**. Compounds that form optical isomers are said to be **chiral**.

The chemistry of enantiomers is of great importance in the field of medicine. It has been

discovered that with many drugs, one enantiomer will be biologically active while the other will be inactive or even produce undesired side effects. For this reason, it has become advantageous for pharmaceutical companies to try to synthesize the active enantiomer exclusively.

The next part of the lab deals with isomers. Isomers are molecules having the same molecular



formula, but the atoms are arranged in a different manner, while still obeying the rules of bonding.

There are different classifications for isomers. For example, **structural isomers** differ from one another in the order in which the atoms are bound to each other (connectivity is different). On the other hand, geometrical isomers have the same order of atoms, but the spatial arrangement of

atoms is different (connectivity is the same). A common example of geometrical isomers is the *cis* and *trans* forms of double bonds:

Figure 5.3.

**** NOTE:** Remember that molecules having single carbon-carbon bonds cannot have *cis/trans* isomers because there is free rotation about single bonds.

By building the models of various molecules during this lab, you will be able to better understand molecular symmetry and isomers. Building models is not difficult; however, the chemical principles involved are very important and you may find some surprises in how atoms can be fit together.

Finally, in Part 4, you will be applying your knowledge of VSEPR (Valence Shell Electron Pair Repulsion) Theory in order to determine the geometry of several different molecules. VSEPR

theory is useful in helping to determine how atoms will orient themselves in molecules. Basically, the idea is that the arrangement adopted by a molecule will be the one in which the repulsions

among the various **electron domains** are minimized. The two kinds of electron domains are

bonding (electron pair shared by two atoms) and non-bonding (electron density centralized on one atom) pairs of electrons.

Experimental Procedure

For Parts 1 & 2: You and your lab partner are to work with one other lab group in preparing these

models (no more than 3 - 4 students). Your TA will assign each group a certain set of molecules to make and answer questions pertaining to those molecules. Each group will then present their answers to the class. These models will need to be completed and answers determined within 30 minutes so that we can continue to Parts 3 & 4 as soon as possible.

For Parts 1-4, the work should be divided among the group members. Be sure to discuss the questions and answers among yourselves, but put your own conclusions on the Report Form.

1. Symmetry Elements

Using the Molecular Framework models, make models of the following compounds:

1. CH₄
2. CH₃Cl
3. CH₂Cl₂
4. CHCl₃
5. CH₂ClF
6. CHBrClF
7. BF₃
8. BF₂Cl
9. PH₃
10. PH₂Cl

Choose a color to represent each atom. For example, make all C atoms black, all H atoms white, etc.

Once the models are created, look for **symmetry elements** that may be present. Ask yourselves the following questions:

Does the molecule contain a **mirror plane (σ)**? In other words, is there a plane within the molecule which results in one half being a mirror image of the other half?

Does the molecule contain a **two-fold rotation axis (C₂)**? Remember from the Introduction that the subscript indicates the degrees of rotation necessary to reach a configuration that is indistinguishable from the original one. In this case, the rotation will be 180°.

Does the molecule contain any higher-order rotation axes?

C₃ – rotation by 120°

C₄ – rotation by 90°

C_∞ (infinity rotation axis) – rotation of any amount will result in an indistinguishable orientation

Does the molecule have an **inversion center (i)**?

Determine which of these symmetry elements are present in your assigned molecules. All of the columns of the table on the report form should be filled out. If you have any difficulty determining whether such symmetry elements are present in the molecules you are assigned, your TA can provide examples of each symmetry element.

Extra credit points can be earned by indicating in the table how many of each symmetry element are present for each molecule (i.e. How many mirror planes are present?).

2. Mirror Images

Using the model kits, build models which are the mirror images of the models you were assigned to build (b, c, d, e, f, g, h, i and j) in Part 1. With the two mirror images in hand, try to place the models on top of one another, atom for atom.

If you can do this, the model and its mirror image are **superimposable** mirror images of one another. If not, the molecule and its mirror image form **nonsuperimposable** mirror images.

Nonsuperimposable mirror images are also known as **enantiomers**.

For each compound, decide whether the mirror image is superimposable or nonsuperimposable.

Can you make a generalization about when to expect molecules to have nonsuperimposable mirror images?

3. Isomers

In this exercise you will build models of compounds which are structural and/or geometrical isomers of one another.

Make the following models:

A. Structural Isomers

1. Make a model(s) of C_2H_5Cl . How many different structural isomers are possible?
2. Make a model(s) of C_3H_7Cl . How many different structural isomers are possible?
3. Make a model(s) of $C_3H_6Cl_2$. How many different structural isomers are possible?

B. Geometrical Isomers

1. Make a model(s) of C_2H_3Cl . How many different structural and geometrical isomers are possible?
2. Make a model(s) of $C_2H_2Cl_2$. How many different structural and geometrical isomers are possible?
3. Make a model(s) of cyclobutane (C_4H_8). HINT: cyclo = ring of C atoms
4. Now make dichlorocyclobutane ($C_4H_6Cl_2$) by replacing two H atoms on cyclopropane with Cl atoms. How many different structural and geometrical isomers are possible for dichlorocyclobutane? You may wish to make a couple of cyclobutane molecules so that you can compare the structures. Do any of the isomers have nonsuperimposable mirror images?

C. Aromatic Ring Compounds

1. Make a model of benzene, C_6H_6 . Even though your model will contain alternating double and single bonds, remember that in the real molecules of benzene all the C-C bonds are equivalent. What symmetry elements does benzene possess?
2. Make a model(s) of chlorobenzene, C_6H_5Cl . How many different structural and geometrical isomers are possible?
3. Make a model(s) of dichlorobenzene, $C_6H_4Cl_2$. How many different structural and geometrical isomers are possible?
4. Make a model(s) of trichlorobenzene, $C_6H_3Cl_3$. How many different structural and geometrical isomers are possible?

4. Hypervalent Structures

Hypervalent compounds are those that have **more than an octet of electrons** around them. Such compounds are formed commonly with the heavier main group elements such as Si, Ge, Sn, Pb, P,

As, Sb, Bi, S, Se, Te, etc. but rarely with C, N or O. Refer to the rules for Electron Domain theory in order to assign Lewis structures to the following molecules. Describe possible isomeric forms and the bond angles between the atoms. How many lone pairs of electrons are present on the

central atom of each molecule, if any? (** Your book will be very useful in aiding you with these structures **)

1. PF₅
2. PF₃Cl₂
3. SF₄
4. XeF₂
5. BrF₃
5. BrF₃

Solutions

Chapter 6. Nanotechnology: Ferrofluids and Liquid

Crystals

Nanotechnology: Ferrofluids and Liquid Crystals

Objective

To synthesize an aqueous ferrofluid (magnetite) and observe its properties.

To understand how nanotechnology affects everyday life.

To learn about surfactants and how they work.

Grading

Pre-Lab (10%)

Lab Report Form (80%)

TA Points (10%)

Background Information

Nanotechnology is the science of controlling matter with dimensions between 1 and 100 nanometers. This includes manipulating individual molecules. It is a multidisciplinary field consisting of physics, biology, chemistry, medicine, engineering, and nearly any other applied science. The prefix nano- means ten to the minus ninth power, or one billionth. There have been great advances in nanotechnology in recent years, and scientists routinely make materials that are only a few nanometers in size, or about 1/80,000 the diameter of a human hair. See Figure 1 to notice how small a nanometer is compared to other common materials.

Materials at the nanoscale exhibit interesting optical, electronic, physical, and chemical properties due to their small size. For example, catalysis chemical reactions occur at the surface of bulk

material so as particles become smaller, the ratio of the surface area to the volume of the particles increases, thereby making a volume of nanoparticle catalysts more reactive than an equal volume

of bulk catalyst. Optical properties of bulk materials are not size dependant, that is no matter what the size of a piece of bulk material it will have the same optical properties. This is not the case for nanomaterials. As you will see in the instructor demo, different sizes of gold nanoparticles exhibit very different colors.

The Scale of Things – Nanometers and More

Things Natural

Dust mite
200 μm

Human hair
~ 60-120 μm wide

Red blood cells with white cell
~ 2-5 μm

Ant
~ 5 mm

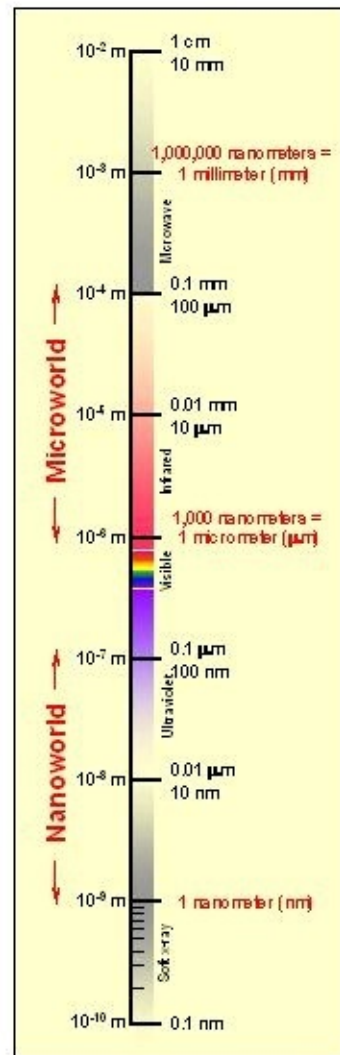
Fly ash
~ 10-20 μm

~ 10 nm diameter

ATP synthase

DNA
~ 2-12 nm diameter

Atoms of silicon spacing ~ tenths of nm



Things Manmade

Head of a pin
1-2 mm

Micro Electro Mechanical (MEMS) devices
10 - 100 μm wide

Pollen grain

Red blood cells

Zone plate x-ray "lens"
Outer ring spacing ~35 nm

Self-assembled,
Nature-inspired structure
Many 10s of nm

Nanotube electrodes

Quantum corral of 48 iron atoms on copper surface
positioned one at a time with an STM tip
Conical diameter 14 nm

Carbon nanotube
~ 1.3 nm diameter

Carbon buckyball
~ 1 nm diameter

The Challenge

Fabricate and combine nanoscale building blocks to make useful devices, e.g., a photosynthetic reaction center with integral semiconductor stripe.

In the 1960s NASA Research Centers discovered fluids that could be controlled through the application of a magnetic field. These fluids were developed to confine liquids in space. These nanoparticle fluids are commonly known as ferrofluids and they are still an active area of research.

Ferrofluids have many current industrial applications. They are used to dampen vibration in audio

loudspeakers, they can behave as liquid O-rings in rotating shaft seals, and they are used in high-speed computer disk drives to eliminate impurities. They also have many potential applications in biomedical, environmental, and engineering fields.

Figure 6.1.

Figure 1-Obtained from Office of Basic Energy Sciences, US Department of Energy

A ferrofluid is a stable colloid suspension of magnetic nanoparticles in a liquid carrier. The nanoparticles are suspended throughout the liquid and have an average size of ~ 10 nm. It is critical that the nanoparticles are coated with surfactant to prevent the particles from aggregating together. The surfactants must be strong enough to prevent agglomeration even when a magnetic field is applied and they must overcome the intermolecular forces between the nanoparticles. For this reason, a typical ferrofluid contains 5% magnetic nanoparticles, 10% surfactant, and 85% carrier fluid by volume.

Figure 6.2.



There are two basic steps in creating a ferrofluid: synthesis of the magnetic solid, magnetite (), and suspension in water with the aid of a surfactant. The magnetic particles must be very small on the order of 10 nm (100 \AA) in diameter, so that the thermal energy of the particles is large enough to overcome the magnetic interactions between particles. If the particles are too large, magnetic interactions will dominate and the particles will agglomerate. The magnetite will be synthesized by a precipitation reaction that occurs upon mixing and with ammonium

hydroxide (an aqueous solution of ammonia,

). The unbalanced equation for this reaction is as

follows:

(6.1)

The surfactant used in this synthesis is tetramethylammonium hydroxide (

). The

hydroxide (

) ions formed in solution tend to bind to the iron sites on the magnetite particles,

creating a net negative charge on each particle. The positively-charged tetramethylammonium

ions will then associate with the negatively-charged magnetite particles, forming a kind of shell around each magnetite particle. This charged shell raises the energy required for the particles to agglomerate, stabilizing the suspension.

Changing the subject to liquid crystals: with the help of nanotechnology, liquid crystal displays have become very popular in recent years. Liquid crystal displays (LCD) were first produced by

RCA in 1971 and are composed of two glass plates with a liquid crystal material between them.

The liquid crystal material is an organic compound that is in a state between a liquid and a solid.

Their viscosities are similar to those of liquids and their light scattering and reflection properties are similar to solid crystals. Liquid crystals must be geometrically highly

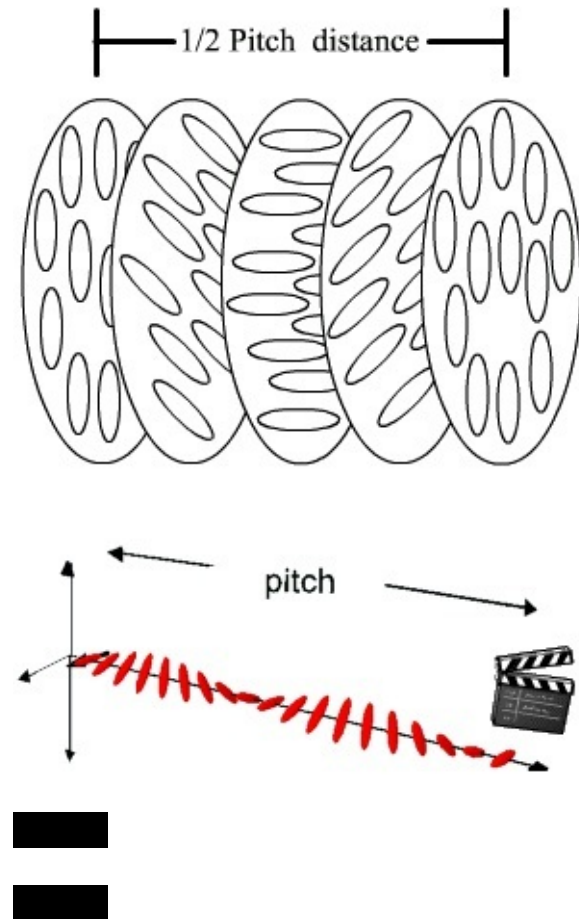
anisotropic (having different optical properties in different directions)-usually long and narrow -

but also become an isotropic liquid (same optical properties in all directions) through a stimulus such as a magnetic field, electric field, temperature, or pressure.

Liquid crystals have several common phases. The simplest liquid crystal phase is called the nematic phase where the molecules spontaneously order with long axes roughly parallel. It is characterized by a high degree of long range orientational order but no translational order. An uniformly aligned nematic has a preferred direction, often described in terms of a unit vector called the director. The type of phase that a liquid crystal possesses ultimately determines its applications.

A subclass of nematic phases that will be investigated in this lab due to its pressure and

temperature sensitive properties is the cholestric phase. The distance over which the director rotates to equal 360° is referred to as the chiral pitch and is normally on the order of a few hundred nanometers, or precisely the wavelength of visible light. This allows liquid crystals to selectively reflect light of wavelengths equal to the pitch length, so that a color will be reflected when the pitch is equal to the corresponding wavelength of light in the visible spectrum. Changes in the director orientation between successive layers modifies the pitch length resulting in an alteration of the wavelength of reflected light according to the temperature. The angle at which the



director changes can be made larger, and thus tighten the pitch, by increasing the temperature of the molecules, hence giving them more thermal energy. Similarly, decreasing the temperature of the molecules increases the pitch length of the chiral nematic liquid crystal. This makes it possible to build a liquid crystal thermometer that displays the temperature of its environment by the reflected color.

Figure 6.3.

Figure 6.4.

Experimental Synthesis

Part I. Synthesis of Gold Nanoparticles (DEMO)

Chemicals

1.0 mM

(stored in amber bottle)

1% trisodium citrate dihydrate solution

Nanoparticle Synthesis

Add 20 mL of 1.0 mM

to a 50 mL Erlenmeyer flask on a stirring hot plate. Add a

magnetic stir bar and bring the solution to a boil.

Add 2 mL of 1% trisodium citrate dihydrate solution and observe the properties.

■

■

■

■

■

■

■

■

Part II. Synthesis of the Aqueous Ferrofluid (Procedure modified from J. Chem.

Edu. 1999, 76, 943-948.)

Chemicals

1 M

in 2 M HCl Solution:

2 M

in 2 M HCl Solution

25% tetramethylammonium hydroxide in water

1.0 M

Solution: Dilute at least 200 mL of concentrated ammonium hydroxide with water to 3.0 L). Open containers of ammonia are odorous and their concentration will decrease over periods of time.

CAUTION: Ferrofluids can be messy. This particular ferrofluid will permanently stain almost all fabrics. Also DO NOT LET THE MAGNETITES TOUCH THE SURFACE OF THE MAGNET DIRECTLY.

Magnetite Synthesis

In a hood, place 4.0 mL of 1M and 1.0 mL of 2M solution into a 100 mL beaker. Stir on a magnetic stir plate.

While stirring, slowly add 50 mL of 1.0 M aqueous solution over a 5 minute period using a buret. Initially a brown precipitate will form followed by a black precipitate, which is magnetite.

CAUTION: Even though 1M is fairly dilute, is a strong base.

Remove from stirring and immediately use a strong magnet to work the stir bar up the walls of the beaker. Remove the stir bar with a gloved hand being careful not to let it touch the magnet.

Allow the magnetite to settle, then decant off the clear liquid into a waste beaker without losing a large amount of precipitate. The settling process can be expedited by placing a strong magnet below the beaker.

Transfer the solid to a plastic weighing boat. Rinse out the beaker with a wash bottle.

Use a strong magnet to attract the ferrofluid to the bottom of the weigh boat. Carefully decant as much clear liquid as possible into the waste beaker. Rinse again with water from the wash

bottle and decant. Repeat the rinsing process a third time. What are you removing by rinsing?

Add 1-2 mL of 25% tetramethylammonium hydroxide. Gently stir the solution with a glass stir

Add 1-2 mL of 25% tetramethylammonium hydroxide. Gently stir the solution with a glass stir

rod for at least a minute to suspend the solid in the liquid. Use a strong magnet to attract the ferrofluid to the bottom of the weigh boat. Pour off and discard the dark liquid. Move the strong magnet around and again pour off any liquid. If the ferrofluid does not produce spikes, continue to move the strong magnet around, pouring off any liquid.

Ferrofluid Properties

1. Hold a magnet underneath the weigh boat that contains the ferrofluid. Move the magnet around the underside of the weigh boat. Move the magnet close to and far from the weigh boat.

Record your observations

2. Add a couple of drops of ferrofluid to a small piece of clean paper. Let the solution dry. Once it is dry, bring a strong magnet close to the paper. What happens?

3. Use several different magnets to observe the properties of the ferrofluid and record your observations in your notebook.

Part III. Synthesis of Cholesteryl Ester Liquid Crystals

Chemicals

Cholesteryl oleyl carbonate

Cholesteryl pelargonate

Cholesteryl benzoate

Vials

Heat gun

Liquid Crystal Synthesis

1. Place 0.65 g cholesteryl oleyl carbonate, 0.25 g cholesteryl pelargonate, and 0.10 g cholesteryl benzoate in a vial.

2. In a hood, with the cap off, melt the solid in with a heat gun.

3. While the mixture is still a liquid, divide it into separate vials using a disposable pipette. Put the caps back on the vials. Allow the vials to cool and observe their properties.

4. Clean up your bench area.

5. Listed below is a chart of the different ratios that produce liquid crystals with different

5. Listed below is a chart of the different ratios that produce liquid crystals with different transition temperatures. Placing liquid crystals with different transition temperatures next to each other on a clear piece of contact paper makes it possible to make a thermometer.

Table 6.1.

Cholesteryl oleyl

Cholesteryl

Cholesteryl

Transition range,

carbonate (g)

pelargonate (g)

benzoate (g)

degrees C

0.65

0.25

0.10

17-23

0.70

0.10

0.20

20-25

0.45

0.45

0.10

26.5-30.5

0.43

0.47

0.10

29-32

0.44

0.46

0.10

30-33

0.42

0.48

0.10

31-34

0.40

0.50

0.10

32-35

0.38

0.52

0.10

33-36

0.36

0.54

0.10

34-37

0.34

0.56

0.10

35-38

0.32

0.58

0.10

36-39

0.30

0.60

0.10

37-40

Solutions



Chapter 7. Solid State and Superconductors

Solid State Structures and Superconductors

Objectives

Build examples of: simple cubic, body centered cubic and face centered cubic cells.

Understand and familiarize with three-dimensionality of solid state structures.

Understand how binary ionic compounds (compounds made up of two different types of ions) pack in a crystal lattice.

Observe the special electromagnetic characteristics of superconducting materials using 1,2,3-
superconductor

, discovered in 1986 by Dr. Paul Chu at the University of Houston.

Grading

Your grade will be determined according to the following

Pre-lab (10%)

Lab report form. (80%)

TA points (10%)

Before coming to lab:

Read introduction and model kits section

Complete prelab exercise

Introduction

From the three states of matter, the solid state is the one in which matter is highly condensed. In the solid state, when atoms, molecules or ions pack in a regular arrangement which can be

repeated "infinitely" in three dimensions, a crystal is formed. A crystalline solid, therefore, possesses long-range order; its atoms, molecules, or ions occupy regular positions which repeat in three dimensions. On the other hand an amorphous solid does not possess any long-range order.

Glass is an example of an amorphous solid. And even though amorphous solids have very

■

■

interesting properties in their own right that differ from those of crystalline materials, we will not consider their structures in this laboratory exercise.

The simplest example of a crystal is table salt, or as we chemists know it, sodium chloride (NaCl).

A crystal of sodium chloride is composed of sodium cations (

) and chlorine anions (

) that

are arranged in a specific order and extend in three dimensions. The ions pack in a way that

maximizes space and provides the right coordination for each atom (ion). Crystals are three

dimensional, and in theory, the perfect crystal would be infinite. Therefore instead of having a molecular formula, crystals have an empirical formula based on stoichiometry. Crystalline

structures are defined by a unit cell which is the smallest unit that contains the stoichiometry and the "spatial arrangement" of the whole crystal. Therefore a unit cell can be seen as the building block of a crystal.

The crystal lattice

In a crystal, the network of atoms, molecules, or ions is known as a crystal lattice or simply as a lattice. In reality, no crystal extends infinitely in three dimensions and the structure (and also properties) of the solid will vary at the surface (boundaries) of the crystal. However, the number of atoms located at the surface of a crystal is very small compared to the number of atoms in the interior of the crystal, and so, to a first approximation, we can ignore the variations at the surface for much of our discussion of crystals. Any location in a crystal lattice is known as a lattice point.

Since the crystal lattice repeats in three dimensions, there will be an entire set of lattice points which are identical. That means that if you were able to make yourself small enough and stand at any such lattice point in the crystal lattice, you would not be able to tell which lattice point of the set you were at – the environment of a lattice point is identical to each correspondent lattice point throughout the crystal. Of course, you could move to a different site (a non-correspondent lattice point) which would look different. This would constitute a different lattice point. For example, when we examine the sodium chloride lattice later, you will notice that the environment of each

sodium ion is identical. If you were to stand at any sodium ion and look around, you would see the same thing. If you stood at a chloride ion, you would see a different environment but that

environment would be the same at every chloride ion. Thus, the sodium ion locations form one set of lattice points and the chloride ion locations form another set. However, lattice points not only exist in atom positions. We could easily define a set of lattice points at the midpoints between the sodium and chloride ions in the crystal lattice of sodium chloride.

The unit cell

Since the crystal lattice is made up of a regular arrangement which repeats in three dimensions, we can save ourselves a great deal of work by considering the simple repeating unit rather than the entire crystal lattice. The basic repeating unit is known as the unit cell. Crystalline solids often have flat, well-defined faces that make definite angles with their neighbors and break cleanly

when struck. These faces lie along well-defined directions in the unit cell.

The unit cell is the smallest, most symmetrical repeating unit that, when translated in three dimensions, will generate the entire crystal lattice.

It is possible to have a number of different choices for the unit cell. By convention, the unit cell that reflects the highest symmetry of the lattice is the one that is chosen. A unit cell may be

thought of as being like a brick which is used to build a building (a crystal). Many bricks are stacked together to create the entire structure. Because the unit cell must translate in three

dimensions, there are certain geometrical constraints placed upon its shape. The main criterion is that the opposite faces of the unit cell must be parallel. Because of this restriction there are only six parameters that we need to define in order to define the shape of the unit cell. These include three edge lengths a , b , and c and three angles α , β and γ . Once these are defined all other distances and angles in

the unit cell are set. As a result of symmetry, some of these angles and edge lengths may be the same. There are only seven different shapes for unit cells possible. These are given in the chart below.

Table 7.1.

Highest Type of Symmetry Element

Unit Cell Type

Restrictions on Unit Cell Parameters

Required

a is not equal to b is not equal to c ; α is

no symmetry is required, an

Triclinic

not equal to β is not equal to γ .

inversion center may be present

a is not equal to b is not equal to c $\alpha =$ highest symmetry element allowed

Monoclinic

$\gamma \neq 90^\circ$ β is not equal to 90° .

is a C_2 axis or a mirror plane

has three mutually

a is not equal to b is not equal to c $\alpha =$

Orthorhombic

perpendicular mirror planes and/or

$\beta = \gamma \neq 90^\circ$

C_2 axes

Tetragonal

$a = b$ is not equal to c $\alpha = \beta = \gamma \neq 90^\circ$

has one C_4 axis

Cubic

$$a = b = c \quad \alpha = \beta = \gamma = 90^\circ$$

has C3 and C4 axes

Hexagonal,

$$a = b \text{ is not equal to } c \quad \alpha = \beta = \gamma = 90^\circ$$

C6 axis (hexagonal); C3 axis

Trigonal

$$\gamma = 120^\circ$$

(trigonal)

Rhombohedral* $a = b = c \quad \alpha = \beta = \gamma$ is not equal to 90°

C3 axis (trigonal)

*There is some discussion about whether the rhombohedral unit cell is a different group or is really a subset of the trigonal/hexagonal types of unit cell.

Stoichiometry

You will be asked to count the number of atoms in each unit cell in order to determine the stoichiometry (atom-to-atom ratio) or empirical formula of the compound. However, it is

important to remember that solid state structures are extended, that is, they extend out in all

directions such that the atoms that lie on the corners, faces, or edges of a unit cell will be shared with other unit cells, and therefore will only contribute a fraction of that boundary atom. As you build crystal lattices in these exercises you will note that eight unit cells come together at a corner. Thus, an atom which lies exactly at the corner of a unit cell will be shared by eight unit cells which means that only $\frac{1}{8}$ of the atom contributes to the stoichiometry of any particular unit cell. Likewise, if an atom is on an edge, only $\frac{1}{4}$ of the atom will be in a unit cell because four unit cells share an edge. An atom on a face will only contribute $\frac{1}{2}$ to each unit cell since the face is shared between two unit cells.

It is very important to understand that the stoichiometry of the atoms within the unit cell must reflect the composition of the bulk material.

Binding forces in a crystal

The forces which stabilize the crystal may be ionic (electrostatic) forces, covalent bonds, metallic bonds, van der Waals forces, hydrogen bonds, or combination of these. The properties of the

crystal will change depending upon what types of bonding is involved in holding the atoms,

molecules, or ions in the lattice. The fundamental types of crystals based upon the types of forces that hold them together are: metallic in which metal cations held together by a sea of electrons, ionic in which cations and anions held together by predominantly electrostatic attractions, and network in which atoms bonded together covalently throughout the solid (also known as covalent crystal or covalent network).

Close-packing

Close-packing of spheres is one example of an arrangement of objects that forms an extended structure. Extended close-packing of spheres results in 74% of the available space being occupied by spheres (or atoms), with the remainder attributed to the empty space between the spheres. This is the highest space-filling efficiency of any sphere-packing arrangement. The nature of extended structures as well as close-packing, which occurs in two forms called hexagonal close packing (hcp) and cubic close packing (ccp), will be explored in this lab activity. Sixty-eight of the ninety naturally occurring elements are metallic elements. Forty of these metals have three-dimensional submicroscopic structures that can be described in terms of close-packing of spheres. Another sixteen of the sixty-eight naturally occurring metallic elements can be described in terms of a different type of extended structure that is not as efficient at space-filling. This structure occupies only 68% of the available space in the unit cell. This second largest subgroup exhibits a sphere packing arrangement called body-centered cubic (bcc).

You should be able to calculate the % of void space using simple geometry.

Packing of more than one type of ion (binary compounds) in a crystal lattice

A very useful way to describe the extended structure of many substances, particularly ionic compounds, is to assume that ions, which may be of different sizes, are spherical. The structure then is based on some type of sphere packing scheme exhibited by the larger ion, with the smaller ion occupying the unused space (interstitial sites). In structures of this type, coordination number refers to the number of nearest neighbors of opposite charge. Salts exhibiting these packing arrangements will be explored in this lab activity.

Coordination number and interstitial sites

When spherical objects of equal size are packed in some type of arrangement, the number of nearest neighbors to any given sphere is dependent upon the efficiency of space filling. The number of nearest neighbors is called the coordination number and abbreviated as CN. The sphere packing schemes with the highest space-filling efficiency will have the highest CN. Coordination

number will be explored in this lab activity. A useful way to describe extended structures, is by using the unit cell which as discussed above is the repeating three-dimensional pattern for

extended structures. A unit cell has a pattern for the objects as well as for the void spaces. The remaining unoccupied space in any sphere packing scheme is found as void space. This void space occurs between the spheres and gives rise to so-called interstitial sites.

Synthesis of solid state materials

There exist many synthetic methods to make crystalline solids. Traditional solid state chemical reactions are often slow and require high temperatures and long periods of time for reactants to form the desired compound. They also require that reactants are mixed in the solid state by

grinding two solids together. In this manner the mixture formed is heterogeneous (i.e. not in the same phase), and high temperatures are required to increase the mobility of the ions that are being formed into the new solid binary phase. Another approach to get solid state binary structures is using a precursor material such as a metal carbonate, that upon decomposition at high

temperatures loses gaseous

resulting in very fine particles of the corresponding metal oxide

(e.g.,

).

X-ray crystallography

To determine the atomic or molecular structure of a crystal diffraction of X-rays is used. It was observed that visible light can be diffracted by the use of optical grids, because these are arranged in a regular manner. Energy sources such as X-rays have such small wavelengths that only “grids”

the size of atoms will be able to diffract X-rays. As mentioned before a crystal has regular

molecular array, and therefore it is possible, to use X-ray diffraction to determine the location of the atoms in crystal lattice. When such an experiment is carried out we say that we have

determined the crystal structure of the substance. The study of crystal structures is known as

crystallography and it is one of the most powerful techniques used today to characterize new

compounds. You will discuss the principles behind X-ray diffraction in the lecture part of this

course.

Superconductors

A superconductor is an element, or compound that will conduct electricity without resistance when it is below a certain temperature. Without resistance the electrical current will flow continuously in a closed loop as long as the material is kept below an specific temperature. Since the electrical resistance is zero, supercurrents are generated in the material to exclude the magnetic fields from a magnet brought near it. The currents which cancel the external field produce magnetic poles opposite to the poles of the permanent magnet, repelling them to provide the lift to [levitate the magnet](#). In some countries (including USA) this magnet levitation has been used for transporation. Specifically trains can take advantage of this levitation to virtually eliminate friction between the vehicle and the tracks. A train levitated over a superconductor can attain speeds over 300 mph!

Solid State Model Kits

In this experiment we will use the Institute for Chemical Education (ICE) Solid-State Model Kits which are designed for creating a variety of common and important solid state structures. Please be careful with these materials as they are quite expensive. There is a list of kit components on the inside of the lid of each box. Please make sure that you have all the listed pieces and that these are in their proper locations when you finish using the kit.

The TAs will deduct points from your lab grade if the kits are not returned with all pieces present and properly organized.

Use of the Solid State Model Kit:

The following instructions are abbreviated. Please consult the instruction manual found in the kits for more details if you need assistance in building any of the structures given. Note that some of the model kits are older than others and the manuals' and page numbers may not correspond.

There are four major part types in each model kit:

*2 off-white, thick plastic template bases with holes (one with a circle, the other a semicircle);

*cardboard templates (about 20 labeled A-T);

*metal rods (to be inserted in the holes to support the plastic spheres)

*plastic spheres in 4 sizes and colors.

The spheres can represent atoms, ions, or even molecules depending upon the kind of solid it is.

You will be given directions for the use of a specific base, template, placement of the rods,

selection of spheres, and arrangement of the spheres as you progress. The ICE model kits make use of Z-diagrams to represent how the structure will be built up. Each type of sphere will be numbered with the z layer in which it belongs.

As we build each structure in three-dimensional space, we will be drawing figures to represent the unit cell structures. Each level or layer of atoms, ions, or molecules in a unit cell can be represented by a two-dimensional base, that is, a square, hexagon, parallelogram, etc.

To draw the Z-diagrams the bottom layer is referred to as $z=0$. We then proceed layer by layer up the unit cell until we reach a layer which is identical to the $z=0$ layer. This is $z=1$. Since $z=0$ and $z=1$ are identical by definition, we do not have to draw $z=1$, although you might want to do so as you are learning how to work with solid state figures. The layers between top and bottom are

given z designations according to their positions in the crystal. So, for example, a unit cell with 4 layers (including $z=0$ and $z=1$) would also have $z=0.33$ ($1/3$) and $z=0.67$ ($2/3$).

Each solid-state kit has two types of bases (one using rectangular coordinates, the other using polar coordinates) indicated by a full circle or semicircle, or by color (yellow and green.)

You will first build structures that involve only one type of atom, as you would find in crystalline solids of the elements, especially that of the metals. Then you will examine ionic compounds

which are known as binary solids. Binary solids are those composed of only two types of atoms, such as sodium chloride or calcium fluoride.

If time permits there is an extra credit exercise you can do. You may not do this extra credit exercise until the report form has been completed nor may you receive credit for the extra credit assignment unless you fully complete the report form.

Working groups and teams

You and your lab partner will constitute a group. Each group will receive one model kit and two

groups will work together as a team. Your TA will assign you the structures you have to do, and at the end each team will discuss the structures assigned on front of the class. The number of teams and the assignments the TA will give you will be decided based on the number of students in a

particular laboratory session. The laboratory is divided for six teams (A-F)

Experimental Procedure

Every part of the experimental procedure has correspondent questions on the Report Form. Do not proceed until ALL questions accompanying each section have been answered and recorded.

1. Demonstration of the 1,2,3-superconductor

A pellet of the 1,2,3-superconductor

is placed on the top of an inverted paper cup. The

pellet is cooled down by carefully pouring liquid nitrogen over it until the bottom of the cup is filled up. After approximately 10 seconds (when the bubbling stops) the pellet should reach the

liquid nitrogen temperature. Your TA will then place a very strong magnet over the pellet.

What happens to the magnet? What happens as the superconductor warms up? What is the

Meissner effect? (Write observations and answer these questions on your report form)

Warning- LIQUID NITROGEN CAN CAUSE FROST BITE! Do not directly touch anything that

has come into contact with the liquid nitrogen until it is warmed up to room temperature.

NOTE TO TA: to remove a levitating magnet, simply wait until the liquid nitrogen fully

evaporates or use another magnet to "grab" the floating magnet. Be careful not to lose or break these very tiny, yet expensive, magnets!!!!

2. Cubic Cells

There are many types of fundamental unit cells, one of which is the cubic cell. In turn, there are three subclasses of the cubic cell:

a. simple or primitive cubic (P)

b. body-centered cubic (bcc, I*)

c. face-centered cubic (fcc, F)

*The I designation for body-centered cubic comes from the German word *innenzentriert*.

We do not have time to build models of all of the unit cells possible, so we will focus on the cubic structure and its variations. Our investigation will include several aspects of each cell type:

the number of atoms per unit cell

the efficiency of the packing of atoms in the volume of each unit cell

the number of nearest neighbors (coordination number) for each type of atom

the stoichiometry (atom-to-atom ratio) of the compound

A. Simple Cubic Unit Cells or Primitive Cubic Unit Cells (P)

Team A

Group 1. Single Unit Cell

- Construct a simple cubic cell using template A and its matching base.
- Insert rods in the 4 circled holes in the shaded region of the template.
- Build the first layer ($z = 0$) by placing a colorless sphere on each rod in the shaded region.
- Draw a picture of this layer as previously described.
- Complete the unit cell by placing 4 colorless spheres on top of the first layer.

This is the $z=1$ layer.

Group 2. Extended Structure

Construct an extended cubic cell using template A.

Insert rods in the circled holes of template A in the area enclosed by the dotted lines.

Construct a set of unit cells as described for making a single unit cell.

Look closely at the structures generated by both groups. They are called simple (or primitive) cubic.

Considering all of the cells around it, answer the corresponding questions on the report form.

B. Body-Centered Cubic Structure (BCC)

Team B

Group 1. Single Unit Cell

- Construct a body-centered cubic (bcc) structure using template F. · Insert the rods in all 5 of the holes in the shaded region.
- Use the guide at left and place four colorless spheres in the first layer (1) at the corners for $z=0$.
- Place one colorless sphere in the second layer (2) on the center rod for $z=0.5$
- Construct the $z=1$ layer.

Group 2. Extended Structure

Using template F, construct an extended body-centered cubic structure.

Insert rods in every hole of the template/block.

Using the guide which follows, place colorless spheres for $z=0$ on every rod labeled 1.



Using the guide which follows, place colorless spheres for $z=0$ on every rod labeled 1.

For $z=0.5$ place colorless spheres on each rod labeled 2.

Complete the $z=1$ layer and then place another two layers on top.

1. Face-Centered Cubic (FCC) Structure

Team C

Group 1. Single Unit Cell

· Construct a single face-centered cubic cell using template C, colorless spheres and the layering as illustrated. Only put rods and spheres on one of the squares defined by the internal lines.

Figure 7.1.

Group 2. Extended Structure

· Construct an extended face-centered cubic structure using template C (You can find instructions on how to do it in the manual that comes with the kit.)

3. Close-Packing: Sphere Packing & Metallic Elements

Team D

Group 1. Construct the hexagonal close-packing unit cell (use the one requiring the C6 template) Group

2. Construct the cubic close-packing unit cell (use the one requiring the C6 template)

Team E

Group 1. Add a 2' layer on top of the existing structure.

Group 2. Add a 2' layer on top of the existing structure.

Team F

Using only the shaded portion on the template, construct the face-centered cubic unit cell which uses the C4 template.

Compare the structures of the face-centered cubic unit cell made on the C4 template to that made on the C6 template.

4. Interstitial sites and coordination number (CN)

Team A

Group 1 - Construct CN 8, CN 6 and CN 4 (using the C4 template).

Group 2 - Construct CN 6, CN 4 (body diagonal) (using the C6 template).

5. Ionic Compounds

Now we will look at some real ionic compounds which crystallize in different cubic unit cells. We will use the models to determine the stoichiometry (atom-to-atom ratios) for a formula unit.

Team B

Cesium Chloride

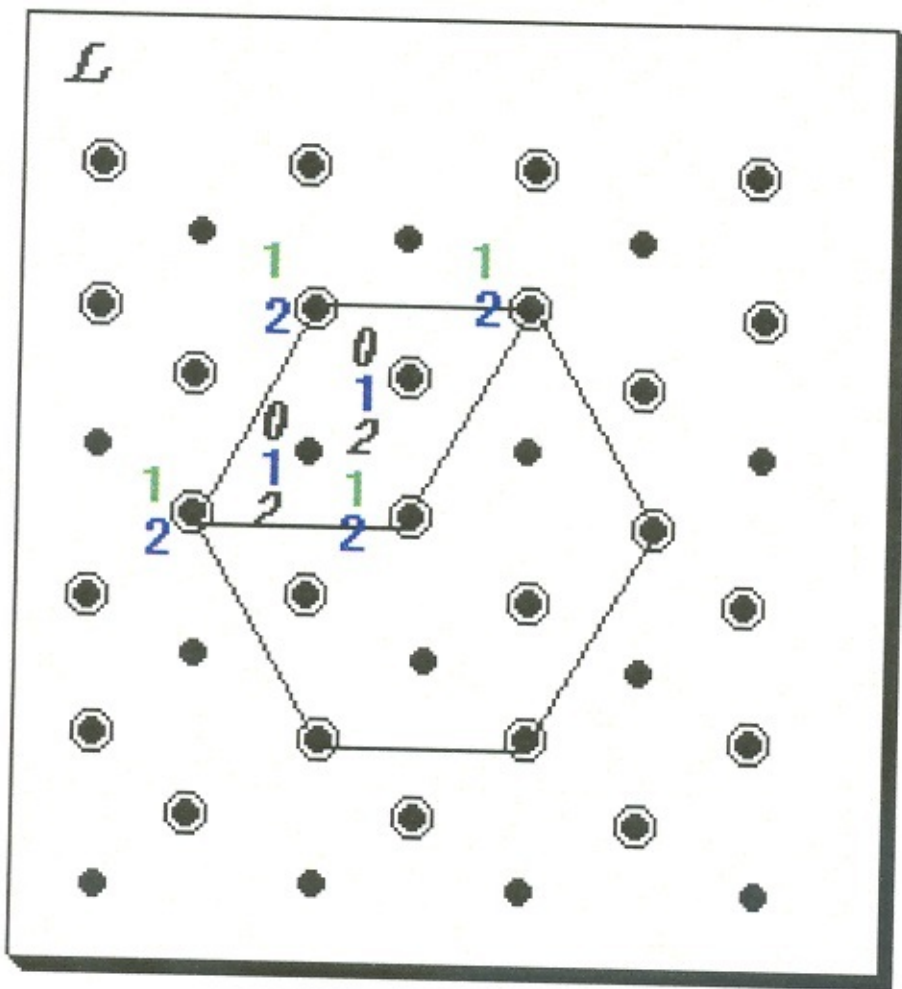
· Construct a model of cesium chloride on template A. This time use colorless spheres as layers 1 and 1' and the green spheres for layer 2.

· Start with the shaded area and then work your way outward to an extended structure. Consider both simple and extended structures when answering the questions which follow.

Team C

Fluorite: Calcium fluoride

· Construct a model of fluorite, which is calcium fluoride, on template E.



- Green spheres will be used for layers 1, 3, and 1' while colorless spheres go on layers 2 and 4.
- Finish with a 1' layer on top. Build the structure by placing rods in all 13 holes in the area enclosed by the internal line.

Team D

Lithium Nitride

- Use the L template and insert 6 rods in the parallelogram portion of the dotted lines.
- Construct the pattern shown below. Be sure to include a $z=1$ layer. 1 is a green sphere while 1 and 2 are blue spheres. The 0 indicates a 4.0 mm spacer tube; the 2 is an 18.6 mm spacer.

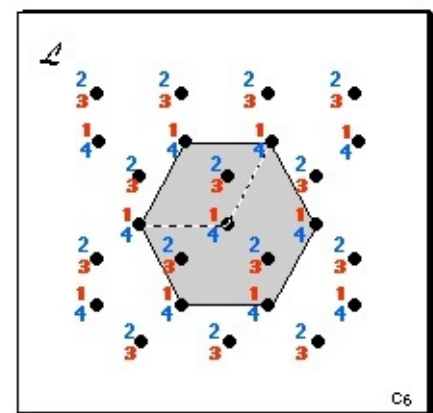
Figure 7.2.

Teams E and F

Zinc Blende and Wurtzite: Zinc Sulfide

Team E. Zinc Blende: Use template D to construct the crystal pattern illustrated below. Numbers 2 and 4 are blue spheres while 1 and 3 are colorless spheres and 4 is a 16.1 mm spacer.

Team F. Wurtzite: Use template L to construct the Wurtzite lattice. Numbers 1, 3 and 1' are



colorless spheres and Numbers 2 and 4 are pink spheres.

Figure 7.3.

Pre-Lab: Solid State and Superconductors

(Total 10 Points)

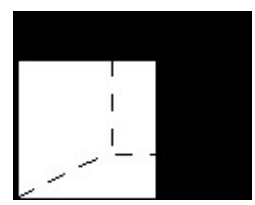
Hopefully [here](#) for the Pre-Lab Name(Print then sign):

Lab Day: _____ Section: _____ TA _____

This assignment must be completed individually and turned in to your TA at the beginning of lab.

You will not be allowed to begin the lab until you have completed this assignment.

1. List the existing crystal systems (unit cell types):
2. Which of these unit cells will we study in this laboratory exercise?
3. Which are the three subclasses of this type of unit cell?
4. Define coordination number:
5. What is the volume of a sphere? Of a cube?



Report: Solid State and Superconductors

Hopefully [here](#) for the Report Form Note: In preparing this report you are free to use references and consult with others. However, you may not copy from other students' work (including your laboratory partner) or misrepresent your own data (see honor code).

Name(Print then sign): _____

Lab Day: _____ Section: _____ TA _____

Part I Demonstration and Unit cell theory

A. TA Demo of the superconductor

Describe and explain your observations (What happens with the magnet? Briefly describe the Meissner effect?)

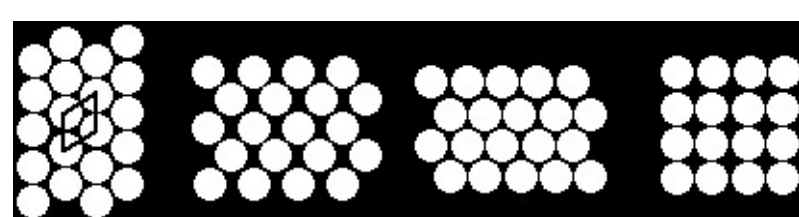
B. The unit cell 1. A cube (see below) has _____ corners, _____ edges & _____ faces.

Figure 7.4.

2. Structure A below shows how a unit cell may be drawn where one choice of unit cell is shown in bold lines. In Structures B, C and D below, draw the outline(s) of the simplest 2-D unit cells (two-dimensional repeating patterns depicted by a parallelogram that encloses a portion of the structure).

If the unit cell is moved in the X,Y-plane in directions parallel to its sides and in distance

increments equal to the length of its sides, it has the property of duplicating the original structural pattern of circles as well as spaces between circles. Can a structure have more than one type of unit cell? _____



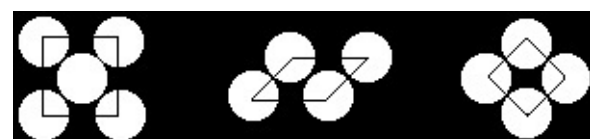


Figure 7.5.

Table 7.2.

Structure A Structure B Structure C Structure D

3. If the circle segments enclosed inside each of the bold-faced parallelograms shown below were cut out and taped together, how many whole circles could be constructed for each one of the

patterns:

Figure 7.6.

Table

7.3.

4. Shown below is a 3-D unit cell for a structure of packed spheres. The center of each of 8 spheres is at a corner of the cube, and the part of each that lies in the interior of the cube is shown. If all of the sphere segments enclosed inside the unit cell could be glued together, how many whole

spheres could be constructed?

number of whole spheres: _____

5. For each of the figures shown below, determine the number of corners and faces. Identify and name each as one of the regular geometric solids.

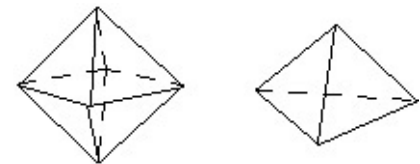


Figure 7.7.

AB

Table 7.4.

A B

Number of corners

Number of faces

Name of the shape of this object

Part II Experimental

1. Cubic Cells

A. Simple Cubic Unit Cells or Primitive Cubic Unit Cells (P)

- a. How would you designate the simple cube stacking - aa, ab, abc, or some other?
- b. If the radius of each atom in this cell is r , what is the equation that describes the volume of the cube generated in terms of r ? (Note that the face of the cube is defined by the position of the rods and does not include the whole sphere.)
- c. Draw the z-diagram for the unit cell layers.
- d. To how many different cells does a corner atom belong? What is the fractional contribution of a single corner atom to a particular unit cell?
- e. How many corner spheres does a single unit cell possess?
- f. What is the net number of atoms in a unit cell? (Number of atoms multiplied by the fraction they contribute)
- g. Pick an interior sphere in the extended array. What is the coordination number (CN) of that atom? In other words, how many spheres are touching it? .
- h. What is the formula for the volume of a sphere with radius r ?
- i. Calculate the packing efficiency of a simple cubic unit cell (the % volume or space occupied by atomic material in the unit cell). Hint: Consider the net number of atoms per simple cubic unit cell (question g) the volume of one sphere (question i), and the volume of the cube (question b).

B. Body-Centered Cubic (BCC) Structure

- a. Draw the z diagrams for the layers.
- b. Fill out the table below for a BCC unit cell

Table 7.5.

Atom type Number Fractional Contribution Total Contribution Coordination Number

Corner

Body

- c. What is the total number of atoms in the unit cell?
- d. Look at the stacking of the layers. How are they arranged if we call the layers a, b, c, etc.?
- e. If the radius of each atom in this cell is r , what is the formula for the volume of the cube generated in terms of the radius of the atom? (See diagrams below.)
- f. Calculate the packing efficiency of the bcc cell: Find the volume occupied by the net number of spheres per unit cell if the radius of each sphere is r ; then calculate the volume of the cube using r of the sphere and the Pythagoras theorem ($a^2 + b^2 = c^2$) to find the diagonal of the cube.

C. The Face Centered Cubic (FCC) Unit Cell

a. Fill out the following table for a FCC unit cell.

Table 7.6.

Atom type Number Fractional Contribution Total Contribution Coordination Number

Corner

Face

- b. What is the total number of atoms in the unit cell?
- c. Using a similar procedure to that applied in Part B above; calculate the packing efficiency of the face-centered cubic unit cell.

1. Close-Packing

- a. Compare the hexagonal and cubic close-packed structures.
- b. Locate the interior sphere in the layer of seven next to the new top layer. For this interior sphere, determine the following:

Table 7.7.

Number of touching spheres:

hexagonal close-packed (hcp) cubic close-packed (ccp)

on layer below

on the same layer

on layer above

TOTAL CN of the interior sphere

c. Sphere packing that has this number (write below) of adjacent and touching nearest neighbors is referred to as close-packed. Non-close-packed structures will have lower coordination numbers.

d. Are the two unit cells the identical?

e. If they are the same, how are they related? If they are different, what makes them different?

f. Is the face-centered cubic unit cell aba or abc layering? Draw a z-diagram.

III. Interstitial sites and coordination number (CN)

a. If the spheres are assumed to be ions, which of the spheres is most likely to be the anion and which the cation, the colorless spheres or the colored spheres? Why?

b. Consider interstitial sites created by spheres of the same size. Rank the interstitial sites, as identified by their coordination numbers, in order of increasing size (for example, which is

biggest, the site with coordination number 4, 6 or 8?).

c. Using basic principles of geometry and assuming that the colorless spheres are the same anion with radius r_A in all three cases, calculate in terms of r_A the maximum radius, r_C , of the cation that will fit inside a hole of CN 4, CN 6 and CN 8. Do this by calculating the ratio of the radius of to cation to the radius of the anion: r_C / r_A .

d. What terms are used to describe the shapes (coordination) of the interstitial sites of CN 4, CN 6 and CN 8?

CN 4: _____

CN 6: _____

CN 8: _____

IV. Ionic Solids

A. Cesium Chloride

1. Fill the table below for Cesium Chloride

Table 7.8.

Colorless spheres Green spheres

Type of cubic structure

Atom represented

2. Using the simplest unit cell described by the colorless spheres, how many net colorless and net green spheres are contained within that unit cell?

3. Do the same for a unit cell bounded by green spheres as you did for the colorless spheres in question 4.

4. What is the ion-to-ion ratio of cesium to chloride in the simplest unit cell which contains both?

(Does it make sense? Do the charges agree?)

B. Calcium Fluoride

1. Draw the z diagrams for the layers (include both colorless and green spheres).

2. Fill the table below for Calcium Fluoride

Table 7.9.

Colorless spheres Green spheres

Type of cubic structure

Atom represented

3. What is the formula for fluorite (calcium fluoride)?

C. Lithium Nitride

1. Draw the z diagrams for the atom layers which you have constructed.

2. What is the stoichiometric ratio of green to blue spheres?

3. Now consider that one sphere represents lithium and the other nitrogen. What is the formula?

4. How does this formula correspond to what might be predicted by the Periodic Table?

D. Zinc Blende and Wurtzite

Fill in the table below:

Table 7.10.

Zinc Blende Wurtzite

Stoichiometric ratio of colorless to pink spheres

Formula unit (one sphere represents and the other the sulfide ion)

Compare to predicted from periodic table

Type of unit cell

Solutions

Chapter 8. Colorful Copper

Experiment 3: Colorful Copper

Objective

To observe, describe and explain the products of a number of chemical reactions of the transition metal copper.

To use several techniques in recovering copper from solution.

To understand the concept of percent yield.

Grading

Pre-lab (10%)

Lab Report (80%)

TA points (10%)

Before Coming to Lab...

Read the lab instructions

Complete the pre-lab, due at the beginning of the lab.

Introduction

Copper is a soft metal with a characteristic color that we often call "copper-colored", a bright orange-brown color. Copper is relatively inert chemically; it does not readily oxidize (react with oxygen) in air and is react when exposed to simple mineral acids such as sulfuric or hydrochloric acid. One of the most popular uses of copper is in the computer industry where it is used to build the integrated circuits and chips. It is beginning to replace aluminum for this application due to the resulting decrease in costs. Copper is also good at conducting electricity because it has so many free electrons that allow for the efficient flow of current.

In this lab you will preform a series of reactions with copper and observe a variety of distinctive and colorful products. Most chemical syntheses involve the separation and then purification of a desired product from unwanted side products. The common methods of separation are filtration,

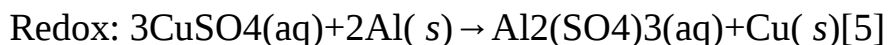
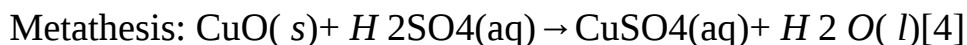
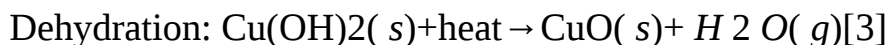
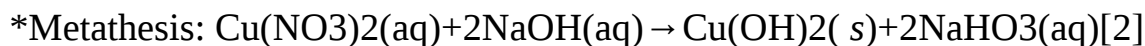
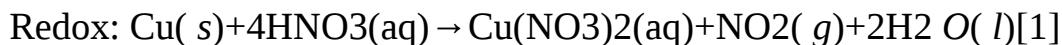
sedimentation, decantation, extraction, chromatography and sublimation.

This experiment is designed as a quantitative evaluation of your laboratory skills in carrying out a series of chemical reactions, purification and analyses with copper. You will employ two

fundamental types of chemical reactions, namely oxidation-reduction (redox) and metathesis

(exchange) reactions to recover pure copper with maximum efficiency. The chemical reactions

involved are the following.



Each of these reactions proceeds to completion and in the case of a metathesis reaction,

completion is reached when one of the components is removed from the solution in form of a gas

or an insoluble precipitate. This is the case for reactions [1], [2], and [3]. In reactions [1] and [3] a gas is formed and in reaction [2] an insoluble precipitate is formed (Reaction [5] proceed to

completion because copper is more difficult to oxidize than aluminum).

Metathesis (Exchange) Reaction Defined in Chapter 4 of your textbook: 'One of the following is

needed to drive a metathesis reaction: the formation of a precipitate, the generation of a gas, the production of a weak electrolyte, or the production of a nonelectrolyte.'

Oxidation-Reduction (Redox) Reactions. This involves the loss of electrons from one components

and an addition of electrons to the other component as the reaction proceeds (the are transferred from one atom to another). The component that loses electrons is said to be oxidized; the one that gains electrons is then reduced. Such reactions are important for the production of electricity due to the energy produced from an electron transfer.

The percent yield of the copper can be expressed as the ratio of the recovered mass to initial mass, multiplied by 100:

$$\% \text{ yield} = (\text{recovered mass of Cu} / \text{initial mass of Cu}) \times 100$$

Experimental Procedure

SAFETY PRECAUTIONS: Wear safety glasses and gloves when handling acids. Work in the fume

hood. Acetone and Methanol are Flammable, so keep them away from flames. Avoid breathing any vapors, especially methanol, as it is very toxic.

1. Preheat a hot plate in the fume hood.
2. Place one strip of fine copper wire (Remember to record the actual weight, approximately 0.5 g) in a 250 mL beaker. Bend the copper wire so that it rests on the bottom of the beaker.
3. Slowly add 30 mL of 6 M HNO₃. Perform this step in the fume hood, as the gas produced is toxic. You may need to gently heat the solution. Observe the color of the gas and solution. Place a wet paper towel and watchglass over the beaker to dissolve the gas. Ask your TA if you are unsure how to do this.
4. Wait until the copper wire dissolves completely before proceeding.
5. Remove beaker from hot plate with a hot hand. Remove watchglass and paper towel. Be sure that the hood is closed as much as possible at this point to avoid breathing in the gas.
6. Add 100 mL of deionized water (Do not use regular water!)
7. Slowly add 30 mL of 6 M NaOH. Note the color of the precipitate and evolution of heat.
8. Add 2 or 3 boiling chips and carefully heat the solution just to boiling point. Note any color change. While waiting for this solution to heat, begin heating ~200 mL deionized water (in a 400 mL beaker) for the next step.
9. Decant some of the supernatant liquid into a 500 mL beaker (Try not to lose any solid while decanting. It is ok to leave some of the liquid behind.). Add about 200 mL of very hot deionized water and allow the precipitate to settle. Decant once more. What are you removing by washing and decantation?
10. Heat the beaker containing the solid for 20 minutes to reduce the volume of the solution (or until the volume has been reduced by half). Having a more concentrated solution will make the following steps proceed faster.
11. Add 15 mL of 6 M H₂SO₄ to the black tarry substance in the beaker (not the solution that you have decanted) while stirring with a glass rod. What copper compound is present now?
12. Remove the boiling chips.
13. In the hood, add 5-10 one-inch squares of aluminum foil and 5-10 ml of concentrated HCl, noting any color changes. STIR WELL. An ideal ratio should be 7 pieces of Aluminum to 10 ml of

acid but you might need more or less depending on the success of your previous steps. If your

solution turns green stop and ask your TA for help. Otherwise, continue to add pieces of aluminum until the supernatant is not blue. Identify what forms on the surface of the aluminum. What is

present in the solution? What gas is formed in the reaction? How do you know?

14. When gas evolution has ceased, decant the solution and transfer the precipitate to a preweighed 100 mL beaker and record its mass on your report form. Wash the precipitated copper with about 5 mL of distilled water and allow it to settle before you decant the solution, and repeat the process. Then wash the precipitate with about 5 mL of methanol. Allow the precipitate to settle, and then decant the methanol. Finally, wash the precipitate with about 5 mL of acetone.

Allow the precipitate to settle again and then decant the acetone from the precipitate. What are you removing by washing?

15. Then use the microwaves to dry your product. Start with 30 seconds and then try 10 seconds more until it is dry. Be sure to let your product cool before weighing it. Then calculate the final mass of copper and from there the weight and percent yield can be determined. Compare the mass with your initial mass and calculate the percent yield. What color is your copper sample in the final step?

Is it uniform in appearance?

Suggest possible sources of error in this experiment.

Solutions

Chapter 9. Metathesis: To Exchange or Not?

Lab 4: Metathesis: To Exchange or Not

Objectives

To give practice writing equations for metathesis reactions, including net ionic equations

To illustrate the concept of solubility and the effect of temperature and crystallization

Grading

You will be determined according to the following:

Pre-lab (10%)

Must attach graph

Lab Report Form (80%)

Must include detailed observations for each reaction

TA Evaluation of lab procedure (10%)

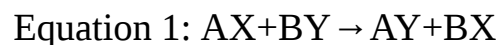
Before Coming to Lab...

Complete the pre-lab exercise, including the plot (due at the beginning of lab)

Read the instructions for the lab and refresh your memory on anything that isn't clear by reading your textbook

Introduction

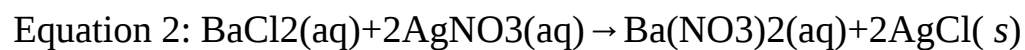
In molecular equations for many aqueous reactions, cations and anions appear to exchange partners. These reactions conform to the following general equation:



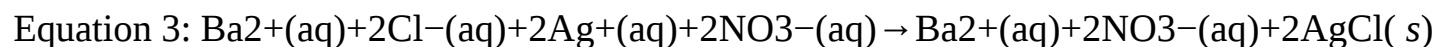
These reactions are known as metathesis reactions. For a metathesis reaction to lead to a net change in solution, ions must be removed from the solution. In general, three chemical processes can lead to the removal of ions from solution, concomitantly serving as a driving force for metathesis to occur:

1. The formation of a precipitate
2. The formation of a weak electrolyte or nonelectrolyte
3. The formation of a gas that escapes from solution

The reaction of barium chloride with silver nitrate is a typical example:

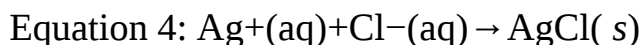


This form of the equation for this reaction is referred to as the molecular equations. Since we know that the salts BaCl_2 , AgNO_3 , and $\text{Ba}(\text{NO}_3)_2$ are strong electrolytes and are completely dissociated in solution, we can more realistically write the equation as follows:



This form, in which all ions are shown, is known as the complete ionic equation. Reaction occurs because the insoluble substance AgCl precipitates out of solution. The other product, barium

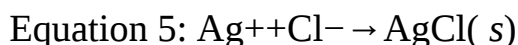
nitrate, is soluble in water and remains in solution. We see that Ba^{2+} and NO_3^- ions appear on both sides of the equation and thus do not enter into the reaction. Such ions are called spectator ions. If we eliminate or omit them from both sides, we obtain the net ionic equation:



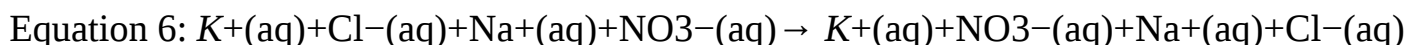
This equation focuses our attention on the salient feature of the reaction: the formation of the precipitate AgCl . It tells us that solutions of any soluble Ag^+ salt and any soluble Cl^- salt, when mixed, will form insoluble AgCl . When writing net ionic equations, remember that only strong

electrolytes are written in the ionic form. Solids, gases, nonelectrolytes, and weak electrolytes are written in the molecular form. Frequently the symbol (aq) is omitted from ionic equations. The

symbols (g) for gas and (s) for solid should not be omitted. Thus, Equation 4 can be written as

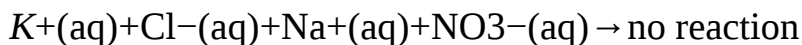


Consider mixing solutions of KCl and NaNO_3 . The ionic equation for the reaction is



Because all the compounds are water-soluble and are strong electrolytes, they have been written in the ionic form. They completely dissolve in water. If we eliminate spectator ions from the

equation, nothing remains. Hence, there is no reaction: Equation 7:

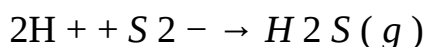


Metathesis reactions occur when a precipitate, a gas, a weak electrolyte, or a nonelectrolyte is formed. The following equations are further illustrations of such processes.

Formation of a Gas

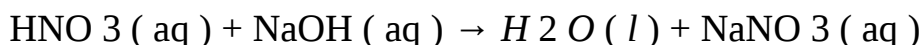
Molecular equation: Equation 8: $2\text{HCl}(\text{aq}) + \text{Na}_2\text{S}(\text{aq}) \rightarrow 2\text{NaCl}(\text{aq}) + \text{H}_2\text{S}(\text{g})$
Complete ionic equation: $2\text{H}^+(\text{aq}) + 2\text{Cl}^-(\text{aq}) + 2\text{Na}^+(\text{aq}) + \text{S}^{2-}(\text{aq}) \rightarrow 2\text{Na}^+(\text{aq}) + 2\text{Cl}^-(\text{aq}) + \text{H}_2\text{S}(\text{g})$
Net ionic equation: $2\text{H}^+(\text{aq}) + \text{S}^{2-}(\text{aq}) \rightarrow \text{H}_2\text{S}(\text{g})$

or



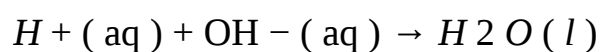
Formation of a Weak Electrolyte

Molecular equation:



Complete ionic equation:

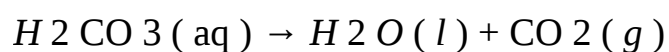
$H^+ (aq) + NO_3^- (aq) + Na^+ (aq) + OH^- (aq) \rightarrow H_2O (l) + Na^+ (aq) + NO_3^- (aq)$ Net ionic equation:



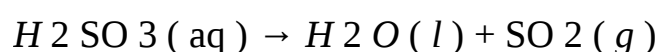
In order to decide if a reaction occurs, we need to be able to determine whether or not a precipitate, a gas, a nonelectrolyte, or a weak electrolyte will be formed. The following brief discussion is intended to aid you in this regard. Table 1 summarizes solubility rules and should be consulted while performing this experiment.

The common gases are CO_2 , SO_2 , H_2S , and NH_3 . Carbon dioxide and sulfur dioxide may be regarded as resulting from the decomposition of their corresponding weak acids, which are

initially formed when carbonate and sulfite salts are treated with acid:



and



Ammonium salts form NH_3 when they are treated with strong bases:

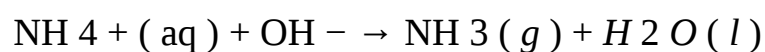


Table 1 Solubility Rules

Table 9.1.

Water-soluble salts

Na^+ , K^+ , NH_4^+

All sodium, potassium, and ammonium salts are soluble.

NO_3^- , ClO_3^- , $\text{C}_2\text{H}_3\text{O}_2^-$ – All nitrates, chlorates, and acetate are soluble.

Cl^-

All chlorides are soluble except AgCl , Hg_2Cl_2 , and

.

Br^-

All bromides are soluble except AgBr , Hg_2Br_2 ,

, and

.

I^-

All iodides are soluble except AgI , Hg_2I_2 , PbI_2 , and HgI_2 .

All sulfates are soluble except

, SrSO_4

SO_4^{2-}

, BaSO_4 , Hg_2SO_4 ,

PbSO_4 , and Ag_2SO_4 .

Table 9.2.

Water-

insoluble salts

CO_3^{2-} , SO_3^{2-} ,

All carbonates, sulfites, phosphates, and chromates are insoluble except those of

PO_4^{3-}

CrO_4^{2-} alkali metals and NH_4^+ .

All hydroxides are insoluble except those of alkali metals and

,

OH^-

, and $\text{Ba}(\text{OH})_2$.

All sulfides are insoluble except those of the alkali metals, alkaline earths, and

S^{2-}

NH_4^+ .

*Slightly soluble.

Table 2 Strong Electrolytes

Table 9.3.

Salts

All common soluble salts

Acids HClO_4 , HCl , HBr , HI , HNO_3 , and H_2SO_4 are strong electrolytes; all others are weak.

Alkali metal hydroxides, $\text{Ca}(\text{OH})_2$

Bases

LiOH , $\text{Sr}(\text{OH})_2$, and $\text{Ba}(\text{OH})_2$ are strong electrolytes; all

others are weak.

Which are the weak electrolytes? The easiest way of answering this question is to identify all of the strong electrolytes, and if the substance does not fall in that category then it is a weak

electrolyte. Note, water is a nonelectrolyte. Strong electrolytes are summarized in Table.2.

In the first part of this experiment, you will study some metathesis reactions. In some instances it will be very evident that a reaction has occurred, whereas in others it will not be so apparent. In the doubtful case, use the guidelines above to decide whether or not a reaction has taken place.

You will be given the names of the compounds to use but not their formulas. This is being done deliberately to give practice in writing formulas from names.

In the second part of this experiment, you will study the effect of temperature on solubility. The effect that temperature has on solubility varies from salt to salt. We conclude that mixing

solutions of KCl and NaNO_3 resulted in no reaction (see Equations 6 and 7). What would happen if we cooled such a mixture? The solution would eventually become saturated with respect to one of

the salts, and crystals of that salt would begin to appear as its solubility was exceeded.

Examination of Equation 6 reveals that crystals of any of the following salts could appear

initially: KNO_3 , KCl , NaNO_3 , or NaCl . Consequently, if a solution containing Na^+ , K^+ , Cl^- , and NO_3^- ions is evaporated at a given temperature, the solution becomes more and more concentrated

and will eventually become saturated with respect to one of the four compounds. If an evaporation is continued, that compound will crystallize out, removing its ions from solution. The other ions will remain in solution and increase in concentration. Before beginning this laboratory exercise you are to plot a graph of the solubilities of the four salts given in Table 3 on your report sheet.

Experimental Procedure

Part 1: Metathesis Reactions

CAUTION WEAR EYE PROTECTION

1. The report sheet lists 16 pairs of chemicals that are to be mixed. Use about 1 mL of the reagents to be combined as indicated on the report sheet.

2. Mix the solutions in small test tubes and record your observations on the report sheet. If there is no reaction, write N.R. (The reactions need not be carried out in the order listed. In order to reduce congestion at the reagent shelf, half the class will start in reverse order). Dispose of the contents of your test tubes in the designated receptacles.

Part 2: Solubility, Temperature and Crystallization

1. Place 8.5 g of sodium nitrate and 7.5 g of potassium chloride in a 100-mL beaker and add 25 mL of water. Warm the mixture on a hotplate, stirring, until the solids completely dissolve.

2. Assuming a volume of 25 mL for the solution, calculate the molarity of the solution with respect to NaNO_3 , KCl , NaCl , and KNO_3 , and record these molarities on your report form.

3. Cool the solution to about 10°C by placing the beaker in ice water in a 600-mL beaker and stir the solution carefully with a thermometer, being careful not to break it.

4. When no more crystals form, at approximately 10°C , filter the cold solution quickly and allow the filtrate to drain thoroughly into an evaporating dish. Dry the crystals between two dry pieces of filter paper or paper towels.

5. Examine the crystals with a magnifying glass (or fill a Florence flask with water and look at the crystals through it). Describe the shape of the crystals—that is, needles, cubes, plates, rhombs, and so forth on your report form.

6. Based upon your solubility graph, which compound crystallized out of solution and write that in the

appropriate place on your report form

7. Evaporate the filtrate to about half of its volume using a Bunsen burner and ring stand. A

second crop of crystals should form. Record the temperature and rapidly filter the hot solution, collecting the filtrate in a clean 100-mL beaker.

8. Dry the second batch of crystals between two pieces of filter paper and examine their shape.

Compare their shape with the first batch of crystals.

9. Based upon your solubility graph, what is this substance?

10. Finally, cool the filtrate to 10°C while stirring carefully with a thermometer to obtain a third crop of crystals. Carefully observe their shapes and compare them with those of the first and

second batches.

11. What compound is the third batch of crystals? Dispose of the chemicals in the designated receptacles.

Pre-Lab 4: Metathesis – To Exchange or Not

Hopefully [here](#) for the Pre-Lab Name(Print then sign):

Lab Day: _____ Section: _____ TA _____

This assignment must be completed individually and turned in to your TA at the beginning of lab.

You will not be allowed to begin the lab until you have completed this assignment.

1. Write molecular, complete ionic, and net ionic equations for the reactions that occur, if any, when solutions of the following substances are mixed: (a) nitric acid and barium carbonate

(b) zinc chloride and lead nitrate

(c) acetic acid and sodium hydroxide

(d) calcium nitrate and sodium carbonate

(e) ammonium chloride and potassium hydroxide

2. Which of the following are not water soluble: Ba(NO₃)₂, FeCl₃, CuCO₃, CuSO₄, ZnS, ZnSO₄?

3. Write equations for the decomposition of H₂CO₃ and H₂SO₃.

4. At what temperature (from your graph) do KNO₃ and NaCl have the same molar solubility?

5. Which of the following are strong electrolytes: BaCl₂, AgNO₃, HCl, HNO₃, HC₂H₃O₂?

6. Which of the following are weak electrolytes: HNO₃, HF, HCl, NH₃(aq), NaOH?

7. For each of the following water-soluble compounds, indicate the ions present in an aqueous solution: NaI, K₂SO₄, NaCN, Ba(OH)₂, (NH₄)₂SO₄.

Report 4: Metathesis – To Exchange or Not

Hopefully [here](#) for the Report Form Note: In preparing this report you are free to use references and consult with others. However, you may not copy from other students' work (including your laboratory partner) or misrepresent your

own data (see honor code).

Name: _____

Section: _____

Part 1: Metathesis Reactions

1. Copper (II) sulfate + sodium carbonate

Table 9.4.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

2. Copper(II) sulfate + barium chloride

Table 9.5.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

3. Copper(II) sulfate + sodium phosphate

Table 9.6.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

4. Sodium carbonate + sulfuric acid

Table 9.7.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

5. Sodium carbonate + hydrochloric acid

Table 9.8.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

6. Cadmium chloride + sodium sulfide

Table 9.9.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

7. Cadmium chloride + sodium hydroxide

Table 9.10.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

8. Nickel chloride + silver nitrate

Table 9.11.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

9. Nickel chloride + sodium carbonate

Table 9.12.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

10. Hydrochloric acid + sodium hydroxide

Table 9.13.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

11. Ammonium chloride + sodium hydroxide

Table 9.14.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

12. Sodium acetate + hydrochloric acid

Table 9.15.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

13. Sodium sulfide + hydrochloric acid

Table 9.16.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

14. Lead nitrate + sodium sulfide

Table 9.17.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

15. Lead nitrate + sulfuric acid

Table 9.18.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

16. Potassium chloride + sodium nitrate

Table 9.19.

Observations

Molecular equation

Complete ionic equation

Net ionic equation

Part 2: Solubility, Temperature and Crystallization

Table 3 Molar Solubilities of NaCl, NaNO₃, KCl, and KNO₃ (mol/L)

Table 9.20.

Compound 0°C 20°C 40°C 60°C 80°C 100°C

NaCl

5.4 5.4

5.5

5.5

5.5

5.6

NaNO₃

6.7 7.6

8.5

9.4

10.4 11.3

KCl

3.4 4.0

4.6

5.1

5.5

5.8

KNO₃

1.3 3.2

5.2

7.0

9.0

11.0

1. Calculate the initial molarities of NaNO₃, KCl, NaCl, KNO₃.

First Batch of Crystals

1. The first batch of crystals is formed at 10°C. Describe these crystals in terms of shape, color, consistency, etc.
2. Use the solubility chart to determine the identity of the crystals. Explain your answer.

Second Batch of Crystals

1. What was the temperature of the filtrate when the second batch of crystals formed?
2. Describe these crystals in terms of shape, color, consistency, etc.
3. Assuming that the first batch of crystals crystallized completely, removing all of its ions from solution, what is the identity of the second batch of crystals? (use your solubility chart and explain your answer)

Third Batch of Crystals

1. The reason that we see a third batch of crystals is that we don't get complete crystallization of the salts in the 1st and 2nd batches of crystals. Assuming there is a small concentration of each ion left in solution after the second batch of crystals is formed, what is the identity of the third batch of crystals according to your solubility chart?
2. Describe these crystals in terms of shape, color, consistency, etc.

BONUS QUESTION:

Describe two different tests that we could use to determine the identity of each set of crystals. To

receive full credit you must give the name of the test, a description of the theory, and describe the results that you would for your proposed identity of each set of crystals. (5 points max.)

Solutions

Chapter 10. Electrochemistry/Alchemy: Molar Mass of

Cu and Turning Cu into Au

Electrochemistry/Alchemy:

Molar Mass of Cu and Turning Cu into Au

Objectives

To learn Faraday's two laws of electrolysis

To relate an electric current to the passage of an amount of electric charge

To discuss electrolysis in molten salts and in aqueous solutions

To determine the molar mass of copper by electrodeposition from an aqueous solution

To copper into gold (please bring to lab with you a penny that is pre-1982)

Grading

You grade will be determined according to the following:

Pre-lab (10%)

Report Form (80%)

TA evaluation of lab procedure (10%) – including bringing a pre-1982 penny to lab

Before Coming to Lab ...

Read and complete the pre-lab

Read the background information

Read and be familiar with the Lab Instructions

Find a pre-1982 penny and bring it with you to lab

Introduction

Electrochemistry describes the interaction between electrical energy and chemical processes.

Electricity continues to intrigue us, as it has since people first observed the sky shattered by bolts of

lightning. Electrochemistry is of great practical value to contemporary living. Consider the number of batteries used for powering the many portable items of pleasure and need – everything from cassette recorders to hearing aids, from calculators to pacemakers. Pure metals are produced from natural ores, inorganic and organic compounds are synthesized, metal surfaces are plated with other metals or coated with paint to enhance their value and utility – all through electrochemistry.

Electricity is a moving stream of electrical charges. This flow, or electric current, can occur as electrons moving through a wire or as ions flowing through an aqueous solution. If the electrons lost and gained in a spontaneous reaction can flow through a wire on their pathway from the substance oxidized to the substance reduced, the energy of the reaction is released as electrical energy. Conversely, a non-spontaneous redox reaction can be driven forward by the introduction into the system of electrical energy from another source. Any device in which either process can occur is called an electrochemical cell.

There are two types of electrochemical cells. The first type generates electrical energy from a spontaneous redox reaction. These are called voltaic or galvanic cells, common household batteries are classic examples. An Italian physicist, Alessandro Volta in 1800 explained that electricity is generated by the connection of two dissimilar metals separated by any moist body (not necessarily organic). A simple voltaic cell, similar to that made by Volta, can be assembled using twelve pennies and twelve nickels (construct a column of alternating pennies and nickels with each coin separated by disk-size pieces of wet filter paper soaked in salt water).

In the second type of electrochemical cell, called an electrolytic cell, a non-spontaneous redox reaction is caused by the addition of electrical energy from a direct current source such as a generator or a storage battery. The process of generating a non-spontaneous redox reaction by means of electrical energy is called electrolysis.

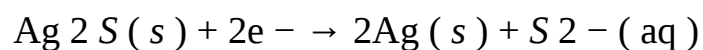
Electrolysis can be used for purifying a metal through the electrolytic dissolution of an impure anode and the subsequent re-crystallization of the pure metal on the cathode. The impurities are left behind in solution. Copper is refined commercially by this electrolytic technique.

Electrolysis is often used for electroplating a metal to another material acting as the cathode. The other material must also be electrically conducting. Non-conducting materials, such as leaves, can also be plated by first being painted with a metallic conductive paint. Silver plating can be done with a silver anode and the object to be plated as the cathode.

Electrolytic reduction (cathodic reduction) has developed into a useful technique for the

restoration of artifacts such as corroded nails and encrusted silver. In the case of silver, the degradation is usually due to the surface formation of insoluble (black) silver sulfide (Ag_2S). The artifact (a silver coin, for example) is attached to the negative electrode of the electrolysis cell.

The Ag^+ ions of the silver sulfide pick up electrons and are converted back to metallic silver:



The sulfide ions are swept away by the water and the surface of the object is restored.

In this experiment, you will electroplate copper quantitatively to a copper cathode (the anode is also composed of copper). The current is measured over an interval of approximately one hour so

that the amount of charge passing through the cell is known. The molar mass of copper is

calculated from its equivalent mass using Faraday's second law. In the second part of the

experiment, you will use turn copper into gold!

Background Information

In the 1830s, Michael Faraday published his experiments using the recently discovered voltaic

column to decompose substances through the use of electric current. Electrolysis is an oxidation-reduction process involving a conversion of electrical energy to chemical energy. The electrolytic cell is a galvanic cell operating in reverse. The automobile battery is acts as a collection of

galvanic cells when delivering electric current, but acts as a collection of electrolytic cells when being recharged.

Faraday first described the quantitative relationships between the amount of electric charge

(number of electrons) that has passed through an electrolytic cell and the amount of materials that have formed at the electrodes. These are summarized as Faraday's Laws of Electrolysis:

1. The mass of substance reacting at an electrode is directly proportional to the total amount of electric charge that has passed through the cell.
2. The masses of the substances reacting at the electrodes are in direct ratio to their equivalent masses. The equivalent mass of a reacting substance is defined as its mass that reacts with one mole of electrons in the oxidation or reduction process. In the case of sodium and chlorine, the equivalent masses of the sodium and chlorine are equal to their molar masses; the equivalent

mass of copper is equal to its molar mass divided by two. The second law is a consequence of

the stoichiometry of the balanced half-reactions.

Through exhaustive experimentation, the charge of a single electron has been determined to be 1.602×10^{-19} coulombs (C). (The coulomb charge unit – defined as useful for much larger charged objects – is inconvenient for expressing such a small charge, so other electrical charge units are commonly used.) One mole of electrons has a total charge calculated to be 96,485 C; this quantity is defined as faraday (F):

$$1 \text{ F} = 96,485 \text{ C/mol e}^-$$

$$n = \frac{120 \text{ C}}{96,485 \text{ C/mole}^-1}$$

Electric currents (I) are measured in amperes (A), amps for short, and defined in terms

$$I = Q/t$$

$$1 \text{ A} = 1 \text{ C/s}$$

For example, a constant current of .600 A (milliamperes) over a period of 2.00×10^2 seconds represents

$$Q = I \times t = 0.600 \text{ A} \times 200 \text{ s} = 0.600 \text{ C/s} \times 200 \text{ s} = 120 \text{ C}$$

a movement of 120 coulombs. The number of moles of electrons (n) transported during the time interval is

Figure 10.1.

$$= 1.24 \times 10^{-2} \text{ mol e}^-$$

Time intervals measured in minutes and hours must be converted to seconds in such calculations.

Another useful equation in Electrochemistry is Ohm's Law, where: $V = I R$ where V is the potential difference in volts, I is the current in amperes and R is a constant, measured in ohms, called the resistance.

Experimental Procedure

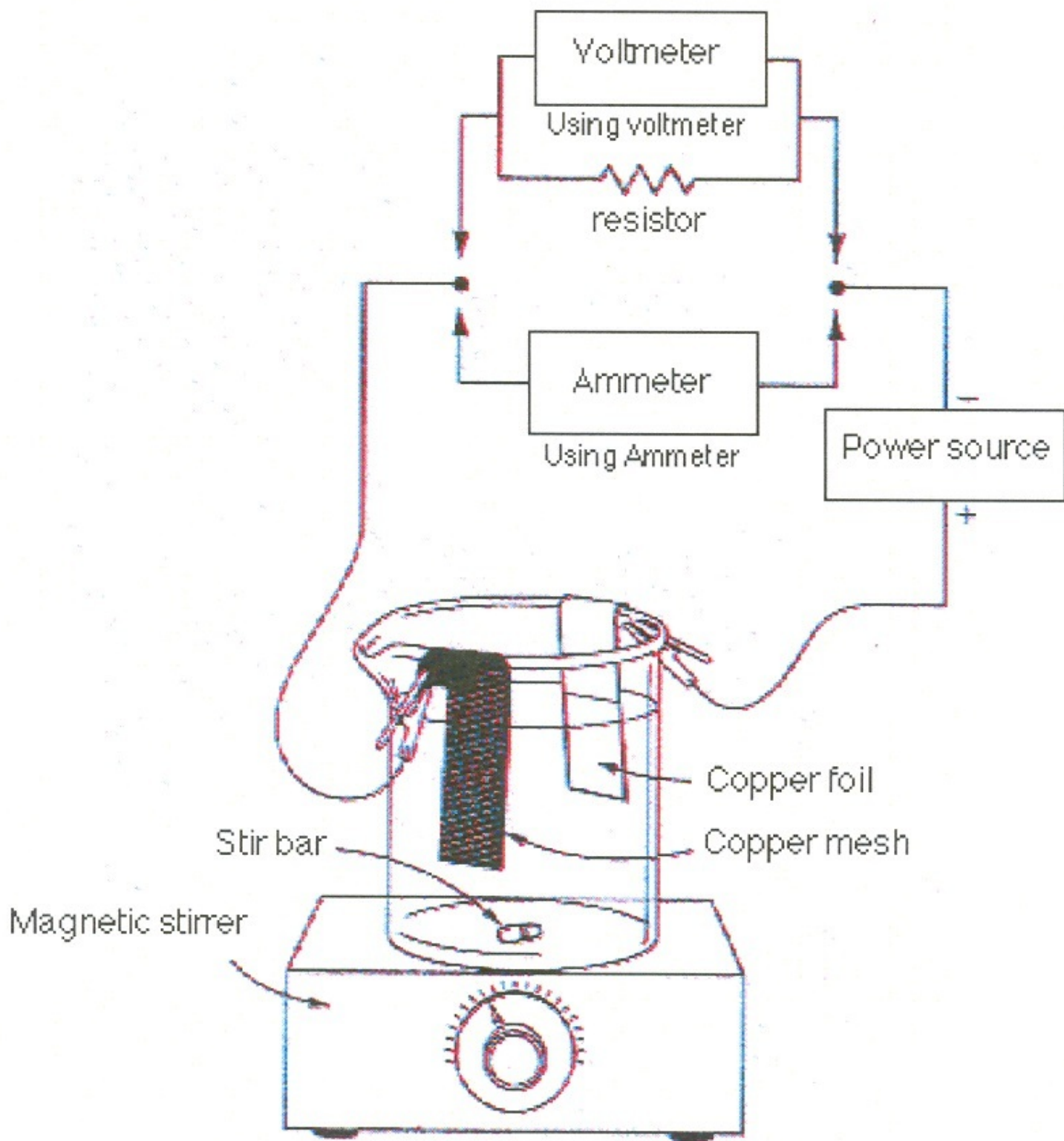


Figure 1

Figure 10.2.

CAUTION WEAR EYE PROTECTION!

CAUTION The 6 M nitric acid used in the next step will burn and stain the skin as well as damage

clothing. In case of skin or clothing contact, wash the area immediately with large amounts of water.

1. Obtain a piece of copper foil (about 2 cm×8 cm). Holding the foil with tweezers or tongs, dip it into 6 M nitric acid several times until its surface is bright and shiny. Do not allow tweezers or tongs to touch the acid solution. Rinse the foil in de-ionized water and set it aside. This is the anode. Set the nitric acid aside to use in the electroplating exercise.

2. Obtain a piece of copper mesh (about 5 cm×8 cm) and remove any loose pieces of copper.

Clean and rinse it as in step 1. Place the copper mesh on a watch glass in the drying oven. Be careful not to touch the cleaned surfaces. This is the cathode.

3. Add 350 mL 1.0 M KNO₃ solution to a 400 mL beaker.

CAUTION The copper sulfate used in the next step is toxic. Avoid skin contact

1. To this solution, add about 5 mL of 1 M H₂SO₄ and 10 g of CuSO₄·5H₂O. Stir until the copper sulfate pentahydrate is fully dissolved.

2. Assemble the apparatus shown in Figure 1, but leave the copper mesh electrode in the oven.

Add a magnetic stirring bar to the beaker. If necessary, add additional 1.0 M KNO₃ to bring solution level in the beaker within 2 cm of the rim. You will either measure the electric current directly with an ammeter in series with the electrolytic cell or you will measure the current indirectly by measuring the voltage across a resistor of known value (about 10 ohms).

3. Remove the copper mesh electrode from the oven, let it cool, and determine its mass to the nearest milligram.

4. Attach the copper mesh electrode to the negative terminal of your power supply using an alligator clip. Turn on the magnetic stirrer.

5. Turn on the low voltage power supply and adjust the current until about 140 mA are flowing through the cell. Record the time and current.

6. Record the time and current every five minutes for an hour.

7. After the last reading, gently remove the cathode from the solution while yet attached to the power supply. After the copper mesh has cleared the solution, remove the wire and turn off the power supply.

8. Gently dip-rinse the copper mesh electrode several times in a beaker of deionized water, and place it on a watch glass in the drying oven.
9. When dry, remove the electrode from the oven and let it cool. Reweigh the mesh electrode.
10. Remove the magnetic stirring bar from your beaker and dispose of the solution in the sink.

Alchemy: Copper into Gold

Place your pre-1982 copper penny in an evaporating dish and heat with a mixture that first turns it silver, then suddenly turns it gold when the penny is then heated on a hot plate.

Caution: Wear safety goggles and gloves, do the reaction in the fume hood with the sash down.

Note step 10: special disposal

1. Place approximately 5 g of zinc in an evaporating dish.
2. Add enough NaOH solution to cover the zinc and fill the dish about one-third.
3. Place the dish on a hot plate and heat until the solution is near boiling.
4. Prepare a copper penny (pre-1982) by cleaning it thoroughly with a light abrasive (steel wool

$$\frac{Q(C)}{96,485C/mol}$$

4. Prepare a copper penny (pre-1982) by cleaning it thoroughly with a light abrasive (steel wool pads work well).
5. Using crucible tongs or tweezers, place the cleaned penny in the mixture in the dish.
6. Leave the penny in the dish for 3-4 min. You will be able to tell when the silver coating is complete.
7. Remove the penny, rinse it, and blot dry with paper towels. (Do not rub.) Remove particles of zinc.
8. Using crucible tongs or tweezers, place the coated penny on the hot plate. The gold color appears immediately.
9. When the gold color forms, remove the coin, rinse it, and dry it with paper towels.
10. Special disposal procedures: Do not discard the waste zinc in the trash container. When zinc dries, it forms a powder that may spontaneously ignite. Rinse the NaOH-zinc mixture several times with water. Then add the solid to a beaker that contains 200 mL of 1 M H_2SO_4 . When all of the solid dissolves, flush the zinc sulfate solution down the drain.

Example of the calculation of Molar Mass:

1. Graph the electric current (in amps) on the y-axis against time (in seconds) on the x-axis. The total charge that passed through the electrolysis cell is given by the area beneath this curve. If the current is constant, this area is:

$$Q = \text{area} = I \times t$$

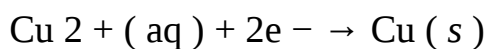
Calculate this charge in coulombs.

1. Convert the coulombs of charge to mol electrons:

$$N =$$

Figure 10.3.

The equation for the reduction half-reaction responsible for the plating at the cathode is



Use the mol ratios of the preceding balanced equation to calculate the number of moles of Cu plated out:

$$\frac{n(\text{Cu})}{n} = \frac{1}{2}$$

$$\frac{m(\text{Cu})}{n}$$

Figure 10.4.

$$n(\text{Cu}) = n/2$$

1. Use the initial and final weighings of the copper mesh electrode to calculate the mass of copper plated out:

$$m(\text{Cu}) = m(\text{final}) - m(\text{initial})$$

1. Calculate the molar mass (M) of copper:

$$M =$$

Figure 10.5.

Electrochemistry Pre-lab Exercise

Hopefully [here](#) for the Pre-Lab Note: In preparing this report you are free to use references and consult with others. However, you may not copy from other students' work (including your laboratory partner) or misrepresent your

own data (see honor code).

Name(Print then sign): _____

Lab Day: _____ Section: _____ TA _____

This assignment must be completed individually and turned in to your TA at the beginning of lab.

You will not be allowed to begin the lab until you have completed this assignment.

1. Write a balanced chemical equation for the electrolysis of molten potassium iodide (KI).
2. An electric current of 0.211 mA passes through an electrolytic cell for 2.00 min. How many moles of electrons have passed through the cell?
3. A voltage drop of 3.412 V is measured across a resistance of 10.51 ohms. How much current is flowing through the resistor?
4. Why is the anode not weighed before electrolysis begins?

Report Form: Electrochemistry

Hopefully [here](#) for the Report Form Note: In preparing this report you are free to use references and consult with others. However, you may not copy from other students' work (including your laboratory partner) or misrepresent your

own data (see honor code).

Name(Print then sign): _____

Lab Day: _____ Section: _____ TA _____

Initial mass of cathode _____ g

Final mass of cathode _____ g

Mass of copper plated out _____ g

Table 10.1.

Time (min) Voltage (V) Current (A)

5

10

15

20
25
30
35
40
45
50
55
60

Show calculations for full credit!

Average Current = (A)

Total charge through cell _____ C

Number of moles e^- through cell _____ mol

Molar mass of copper _____ g/mol

Questions:

1. What is oxidized and what is reduced when electroplating copper?
2. Write the half reactions that occur at the anode and electrode.
3. Why is it important not to touch the cleaned cathode?

4. What is the purpose of KNO_3 in this experiment?
5. How would your results for the molar mass of copper be affected if hydrogen gas were also observed at the cathode?
6. What part of this procedure limits the accuracy of the molar mass determination?

Alchemy - Copper into gold

1. Is this reaction an oxidation-reduction reaction?
1. Why did the penny turn "silver"?
1. Why did it turn "gold"?
1. Why did we heat the penny to turn it "gold"?

Solutions

Chapter 11. Bonding 07

Lab 5: Bonding 07

Objective

To test various compounds and determine their conductivity and bonding.

To understand how electronegativity can predict bond type.

To learn the relationship between bonding and conductivity.

Grading

Pre-Lab (10%)

Lab Report Form (80%)

TA Points (10%)

Background Information

A chemical bond is a link between atoms that results from the mutual attraction of their nuclei for electrons. Bonding occurs in order to lower the total potential energy of each atom or ion.

Throughout nature, changes that decrease potential energy are favored.

The main types of bonds that we will be covering are ionic bonds, covalent bonds, and metallic

bonds. An ionic bond is the chemical bond that results from the electrostatic attraction between positive (cations) and negative (anions) ions. The ionic relationship is a “give and take” relationship. One ion donates or “gives” electrons, while the other ion receives or “takes” electrons.

A covalent bond is a chemical bond resulting from the sharing of electrons between two atoms.

There are two main types of covalent bonds. The first being non-polar covalent bonds. These are bonds in which the bonding electrons are shared equally by the united atoms-with a balanced electrical charge. Polar covalent bonds are covalent bonds in which the united atoms have an unequal attraction for the shared electrons.

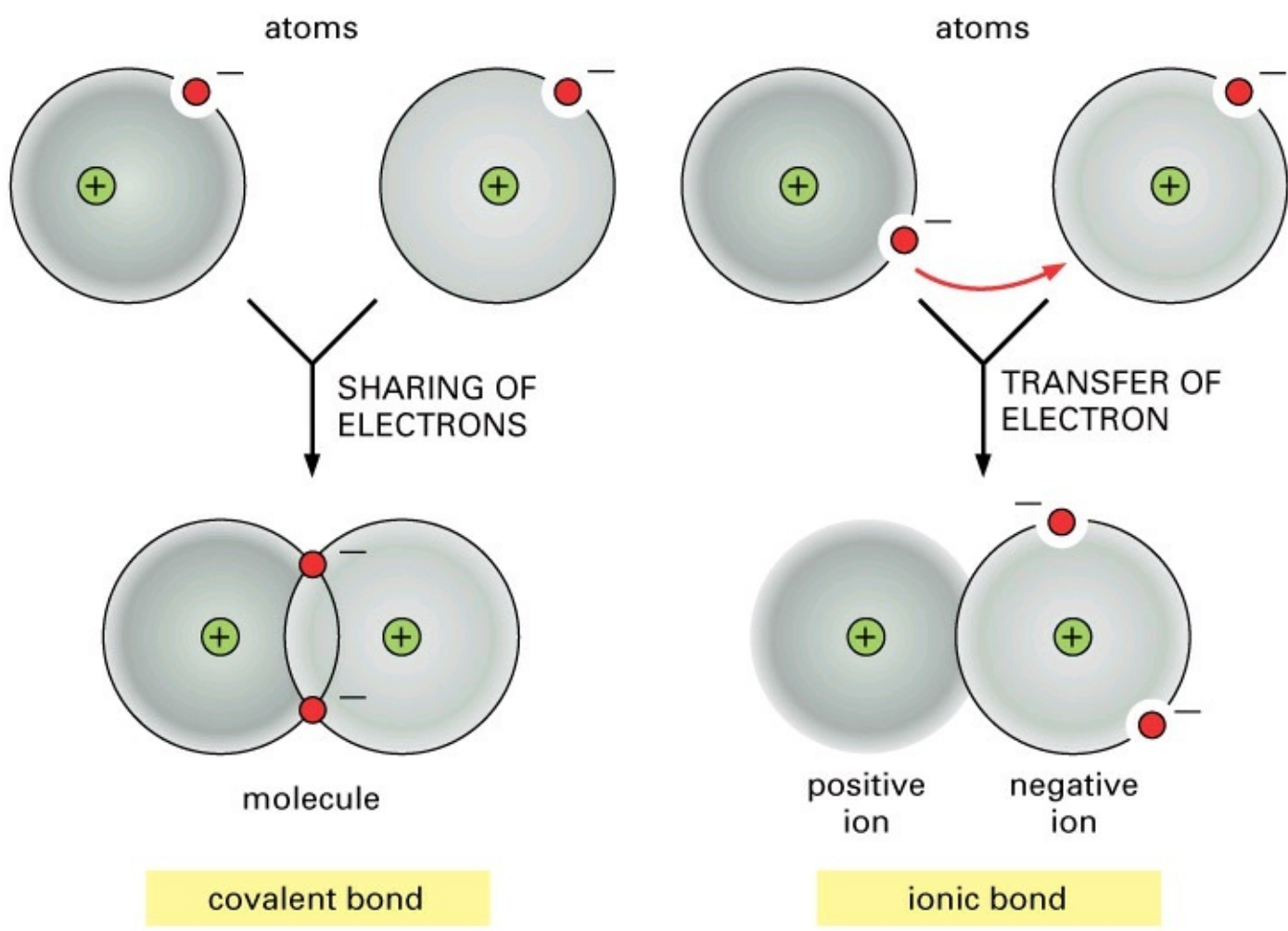


Figure 2.6 Essential Cell Biology, 2/e. (© 2004 Garland Science)

Figure 11.1.

The role of electrons in bonding has been well-studied. The ability of an atom or element to attract electrons to itself is known as the element’s electronegativity. A scale was first calculated by the Nobel laureate Linus Pauling and is commonly called the Pauling electronegativity scale. The

actual electronegativity values aren't as important as how they compare to a different element. In Part I of today's experiment, you will compare electronegativity values to predict the type of bond that will exist between two elements.

In the solution state, ionic compounds dissociate to give a separation of charge. The separation of charge allows for the flow of electrons through solution. The flow of electrons is classified as conductivity. A strong electrolyte is a compound that when dissolved in water will completely

ionize or dissociate into ions. That is, the compound exists in water only as individual ions, and there are no intact molecules at all. This solution conducts electricity well. A weak electrolyte is a compound that when dissolved in water only partially ionizes or dissociates into ions. That is, the compound exists in water as a mixture of individual ions and intact molecules. This solution conducts electricity weakly. A nonelectrolyte is a compound that when dissolved in water does not ionize or dissociate into ions at all. In water, this compound exists entirely as intact molecules.

The solution does not conduct electricity at all. By measuring the conductivity of a dissolved compound, we can classify it as a nonelectrolyte, weak electrolyte, or strong electrolyte and determine its ability to dissociate into ions. There are four common compounds that you will encounter in today's lab.

ACIDS are molecular compounds which ionize (turn into ions) in water. The cation that is formed is always H^+ . Therefore, in the formulas for simple acids, H is always the first element listed.

Some acids are strong electrolytes and some acids are weak electrolytes. There are no acids which are nonelectrolytes because by definition an acid is a H^+ donor.

BASES can be molecular compounds or ionic compounds. Some bases are soluble and some are not. The soluble bases ionize or dissociate into ions in water, and the anion formed is always

OH^- . The ionic bases have hydroxide (OH^-) as the anion. If they are soluble, the ions simply separate (dissociate) in the water. All of the ionic bases which are soluble are strong electrolytes.

SALTS are ionic compounds which are not acids or bases. In other words, the cation is not hydrogen and the anion is not hydroxide. Some salts are soluble in water and some are not. All of the salts which are soluble are relatively strong electrolytes.

NONELECTROLYTES are compounds which dissolve in water but do not ionize or dissociate into ions. These would be molecular compounds other than the acids or bases already discussed.

Experimental Procedure

Caution: Acids and bases are corrosive and can cause burns.

Part I. Predicting bond type through electronegativity differences.

Using the electronegativity table provided in the lab manual, predict the type of bond that each of the following compounds will have by the following process:

Find the electronegativity for each element or ion in compound using electronegativity table provided.

Subtract the electronegativities (using absolute value).

If values are between:

4.0-1.7---Ionic bond-50-100% ionic

1.7-0.3---Polar Covalent bond-5-50% ionic

0.3-0.0---Non-Polar Covalent-0-5% ionic

Determine the type of bonding in the following compounds: KCl, CO, CaBr₂, SiH₄, MgS.

Increasing Electronegativity

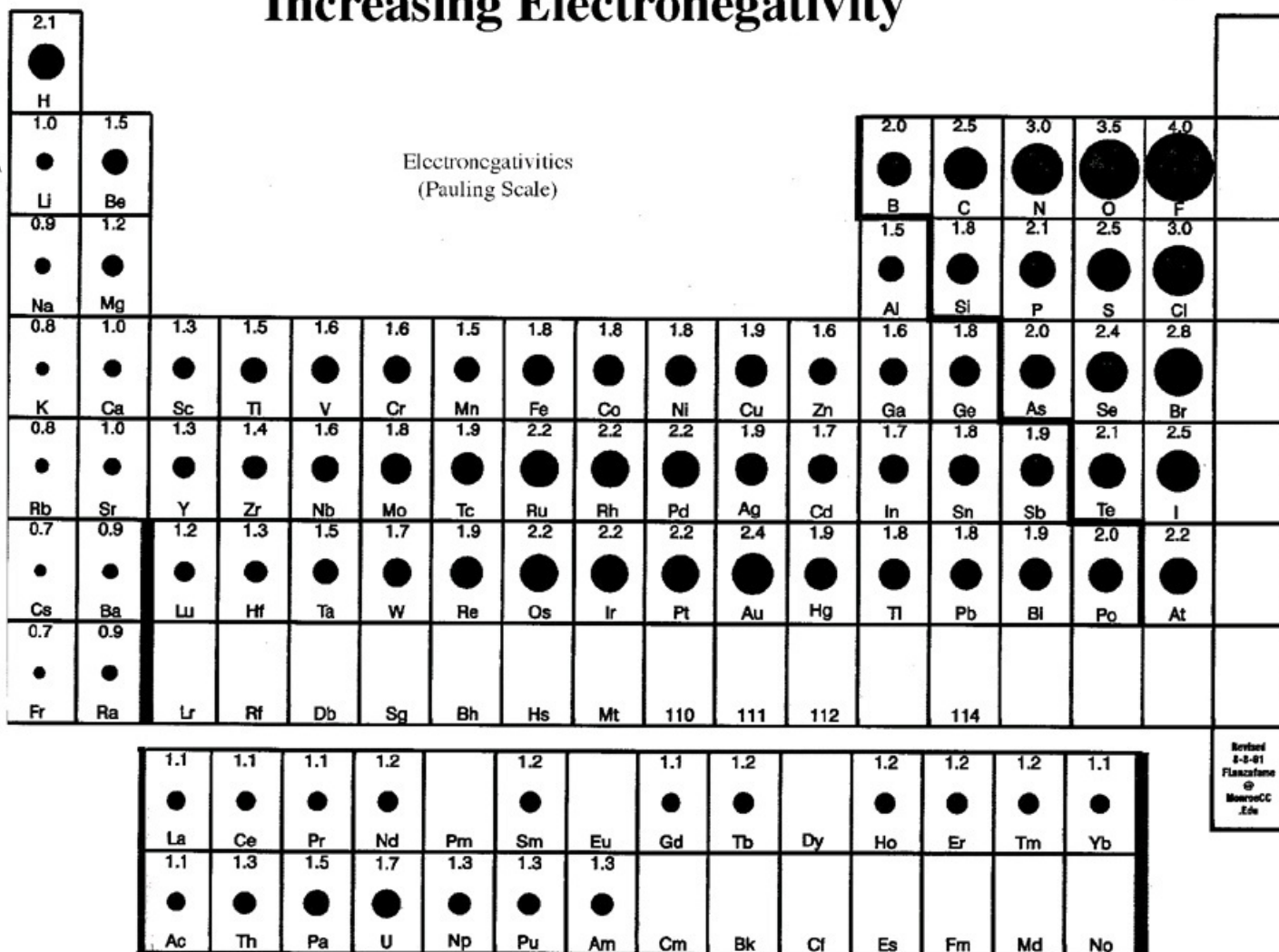


Figure 11.2.

Part II. Weak and strong electrolytes

Chemicals

tap water

0.1 M hydrochloric acid, HCl

0.1 M acetic acid, HC₂H₃O₂

0.1 M sulfuric acid, H₂SO₄

0.1 M sodium hydroxide, NaOH

0.1 M ammonia, NH₃

0.1 M sodium acetate, $\text{NaC}_2\text{H}_3\text{O}_2$

0.1 M sodium chloride, NaCl

0.1 M ammonium acetate, $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$

0.1 M ammonium chloride, NH_4Cl

methanol, CH_3OH

ethanol, $\text{C}_2\text{H}_5\text{OH}$

sucrose solution, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$

In today's lab, you will be using a MicroLab conductivity probe to determine how well electrons flow through a given solution. First, you will need to calibrate the probe with a non-electrolyte (distilled water) and a very strong electrolyte. To quantify how well a solution conducts, we will assign numerical values to the conductance probe. A non-conducting solution will have a conductance value of 0, a poor conducting solution will have a reading of 0 to 1,000, and good conductors will have readings of 3,000 up.

Instructions for MicroLab Conductivity Experiment

Open the MicroLab Program by clicking on the Shortcut to MicroLab.exe tab on the desktop.

On the "Choose an Experiment Type" Tab, enter a name for the experiment, and then double click on the MicroLab Experiment icon

Click "Add Sensor", Choose sensor = Conductivity Probe

Choose an input, click on the red box that corresponds to the port that your conductivity sensor is connected to. Choose 20,000 microseconds

"Choose a Sensor", click radial button that says Conductivity Probe. Click next.

Click "Perform New Calibration"

Click "Add Calibration Point" place the conductivity probe in the non-conductive standard solution, while swirling wait until the value is constant and then enter 0.0 into the "Actual Value" box in MicroLab and hit "ok".

Again, Click "Add Calibration Point" place the conductivity probe in the conducting standard solution, while swirling wait until the value is constant and then enter 1020 into the "Actual

Value” box in MicroLab and hit “ok”. Repeat for 3860 as the Actual Value.

Under Curve Fit Choices , click on “First order (linear)” and then “Accept and Save this

Calibration”, when prompted to “Enter the units for this calibration”, leave as is and click ok, save as your name-experiment-date. Click finish.

In the sensor area, left click on the conductivity icon and drag it to the Y-axis over “data source two”, also click and drag to column B on the spreadsheet and also click and drag to the digital display window.

When ready to obtain data, click start.

This is very important: Be sure to thoroughly rinse the probe with DI water between every use.

Beginning with the tap water, measure the conductance of each of the following solutions. Using the information provided in the lab manual, classify each solution as a non-, weak, or strong electrolyte. For those solutions that are electrolytes, record the ions present in solution.

Part III. Electrolyte strength and reaction rate

Chemicals

calcium carbonate powder - shake once

1 M HCl - stopper it

1 M $\text{HC}_2\text{H}_3\text{O}_2$

0.5 M H_2SO_4

Test tube gas collection apparatus - end at 20mL

Measure 2 g of powdered calcium carbonate (CaCO_3) onto a piece of weigh paper. Obtain 30 mL of 1 M HCl in a graduated cylinder. Pour the acid into the test tube apparatus. Add the calcium carbonate to the acid and QUICKLY stopper the tube to begin collecting gas. Record the time it takes to collect 20 mL of gas. The acid may react very fast with the CaCO_3 generating the gas very rapidly. Clean out the test tube apparatus and repeat the experiment using 1 M $\text{HC}_2\text{H}_3\text{O}_2$ and 0.5 M H_2SO_4 .

Part IV. Chemical reactions

Chemicals

0.01 M calcium hydroxide, $\text{Ca}(\text{OH})_2$

Plastic straws

Plastic straws

Obtain ~20 mL of saturated calcium hydroxide solution. Make sure it is clear and colorless. Place the conductivity probe in the solution and begin monitoring its conductivity. With your straw,

slowly exhale into the solution. Note any observations in the solution and the conductivity.

Pre-Lab 5: Bonding 07

(Total 10 Points)

Hopefully [here](#) for the Pre-Lab Name(Print then sign):

Lab Day: _____ Section: _____ TA _____

This assignment must be completed individually and turned in to your TA at the beginning of lab.

You will not be allowed to begin the lab until you have completed this assignment.

Part I. Bonding of chemicals in solution

1. Write out the formulas of the following acids:

phosphoric _____

perchloric _____

nitric _____

sulfuric _____

hydrochloric _____

acetic _____

1. Write out the formulas of the following bases:

calcium hydroxide _____

potassium hydroxide _____

sodium hydroxide _____

ammonia _____

1. Write out the formulas of the following salts:

potassium chromate _____

potassium sulfate _____

copper(II) nitrate _____

calcium carbonate _____

potassium iodide _____

Report 5: Bonding 07

Hopefully [here](#) for the Report Form Note: In preparing this report you are free to use references and consult with others. However, you may not copy from other students' work (including your laboratory partner) or misrepresent your

own data (see honor code).

Name(Print then sign): _____

Lab Day: _____ Section: _____ TA _____

Part I. Predicting bond type through electronegativity differences.

Table 11.1.

Chemical Formula	Electroneg (1)	Electroneg (2)	Diff Electroneg	Type of bond
------------------	----------------	----------------	-----------------	--------------

KCl

CO

CaBr₂

SiH₄

MgS

Part II. Weak and strong electrolytes

Table 11.2.

Solution Tested	Numerical Output	Electrolyte Strength	Ions Present
-----------------	------------------	----------------------	--------------

0.1 M HCl

0.1 M $\text{HC}_2\text{H}_3\text{O}_2$

0.1 M H_2SO_4

0.1 M NaOH

0.1 M NH_3

0.1 M $\text{NaC}_2\text{H}_3\text{O}_2$

0.1 M NaCl

0.1 M $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$

0.1 M NH_4Cl

CH_3OH

$\text{C}_2\text{H}_5\text{OH}$

Sucrose

Tap water

1. Why do we use deionized water instead of tap water when making solutions for conductivity measurements?

Part III. Electrolyte strength and reaction rate

2. Time to collect 20 mL of gas using 1 M HCl _____ . Write the reaction of HCl with CaCO_3 .

3. Time to collect 20 mL of gas using 1 M $\text{HC}_2\text{H}_3\text{O}_2$ _____. Write the reaction of $\text{HC}_2\text{H}_3\text{O}_2$ with CaCO_3 .

4. Time to collect 20 mL of gas using 0.5 M H_2SO_4 _____. Write the reaction of H_2SO_4 with CaCO_3 .

5. Why does it take different lengths of time to collect 20 mL of gas?

6. Based on the time it took to collect 20 mL of gas, rank the acids in the order of increasing strength.

7. Why did we use 0.5 M H_2SO_4 instead of 1.0 M H_2SO_4 ?

Part IV. Chemical reactions

8. Write the chemical reaction for calcium hydroxide with your exhaled breath.
9. Write your observations for the reaction that took place (i.e. appearance, conductivity, etc.)
10. When in separate solutions, aqueous ammonia, $\text{NH}_3(\text{aq})$ and acetic acid $\text{HC}_2\text{H}_3\text{O}_2$ conduct electricity equally well. However, when the two solutions are mixed a substantial increase in electrical conductivity is observed. Explain.
11. Separately, ammonium sulfate and barium hydroxide solutions are very good conductors. When the two solutions are mixed a substantial decrease in conductivity is observed. Rationalize this.

Solutions

Chapter 12. Silver Nanoparticles: A Case Study in

Cutting Edge Research

Alvin Orbaek, Mallam Phillips, Dr. Mary McHale, Prof. Andrew Barron,

Objective

To gain an insight into nanotechnology, what it is and how it can be useful, using silver nanoparticles as an example. We will look at what exactly nanoparticles are, see how they are made, and how they can be characterized.

The characterization technique involves Ultra-Violet and Visible spectroscopy, so we will look briefly into the interaction of the nanoparticles and light, which will hopefully help you gain an appreciation for one of the special aspects of nanotechnology.

When making the nanoparticles we will do a time study allowing us to graph the spectroscopic response - which will show the nature of the particle as it grows, i.e., ripens. We can use some data to calculate the size of the nanoparticle at the beginning and at the end of our experiment.

Background

What is nanotechnology?

Nano is the ancient Greek word for dwarf. In scientific terms it has been used to identify length scales that are one billionth of a unit. This is typically a meter and so you often here things that are nanometers in size. In terms of nanotechnology it has been defined as anything that has a

unique property or function resulting from the size of the artifact being in the nano regime, and that the size regime is between 0.1 and 100 nm. This size range is rather broad; encompassing

simple molecules to more complicated molecules like enzymes. However, these items can be

looked at from many points of view, from a chemist that considers molecules, to that of an engineer that would look at how each of the molecules interacts in the bigger system and creates new materials from these building blocks. For this reason there are many disciplines that are interested in the study of nanotechnology such as Chemistry, Physics, Engineering, Biological sciences, Material Sciences, Computer Science and many more besides. For this reason nanotechnology is not a strict discipline and many people use their skills and backgrounds from other areas to contribute to research in this particular field.

Why care about nanotechnology?

There are many effects that occur at the nanoscale that we do not notice on a larger macro scale.

Most of nature actually works at the nanoscale, and by understanding the forces that are at work using knowledge from chemistry, physics and engineering one can better understand the working of organic life. Enzymes are very large molecules that are too large to consider in terms of chemistry alone, other effects come into play In order to understand the full picture we need to borrow from physics and computer modeling to gain a better understand of what is happening.

There are many effects that occur at the nanoscale that we do not notice on a larger macro scale.

Most of nature actually works at the nanoscale, and by understanding the forces that are at work using knowledge from chemistry, physics and engineering one can better understand the working of organic life. Enzymes are very large molecules that are too large to consider in terms of chemistry alone, other effects come into play In order to understand the full picture we need to borrow from physics and computer modeling to gain a better understand of what is happening.

When we cross from the small scale as in molecules and atoms, to the large scale that we see with our own eyes, we travel through the nanoscale. In that scale we go from quantum physics to

classical physics and a lot of very interesting effects can be used to our benefit, and actually nanoparticles are an excellent example of this. Just by virtue of their size they are able to absorb four times more light than is even shone on them! This is very different from the bulk material, it is difficult to understand in one sitting, but let's just say that there is a coupling between the light energy and the matter of the nanoparticles that is best explained through quantum mechanics, but we won't go into that now.

When you make something very large, there is lots of room for error, the more parts you have in a system the more chances there are that some of those parts can be faulty. However when you make something in the nanoscale you have far less parts in the system and each part has to be virtually

perfect. Material scientists are concerned with the defects that are created in materials, because these are the parts that cause a material to break down often and stop functioning correctly. As you get into the nanoscale there are less defects and you get enhanced effects from the purer

material, that don't occur on the larger scale. One example of this is carbon nanotubes, by virtue of their shape and size they are 6 times lighter than steel, but almost 100 times stronger. There is great potential for using these in new materials in the future that are ultra lightweight and

extremely strong.

When we make things with modern technology we have for centuries been using a top down approach, and this brings us down to a fine limit but not as fine as that on which nature works.

Nanotechnology is more about understanding the fundamental forces in nature by physics, and seeing their interaction through chemistry, and then making something larger from our

engineering skills. And we can always take examples from biology that has been doing this for far longer than we have. So really what we do is take a bottom up approach, so that we can create

large materials that we can use, that has every part of the interaction tailored all the way from how the atoms interact and how the molecules are formed and bonded together to make building blocks

for new materials and applications. This bottom up approach is a change in the way things have

been done and for this reason nanotechnology is a very potent discipline, with an immense capacity for expansion.

In all we have only really begun to scratch the surface of what could be possible when we create things using nanotechnology, and we should be aware of this because nanotechnology is finding

its way into every corner of life, from health studies, medicine, robotics, materials and maybe

even food and many many more.

What are nanoparticles and how are they made?

A simple way of seeing this is by imagining tennis balls that are squeezed down to a few billionths of a meter. The particles are rounded because they try to minimize the surface energy as much as possible; any edges will make things more energetic since typically nature follows the path of

least resistance the particles tend to form colloids, or spheres with as few edges as possible. It is possible though, to direct the growth of nanoparticles into various shapes such as cubes, and

tetrahedrons. We will concern ourselves with only colloidal nanoparticles for the moment.

The nanoparticles have a large surface area compared with the total volume. The surface area to

volume ratio is interesting because chemical reactions typically occur on surfaces, so

nanoparticles that have a high surface to energy ratio can be used in many interesting ways, such as in catalysis. One teaspoon of nanoparticles might weigh only 200 mg, but because of their

shape and the large amount of surface area the tea spoon could have the same surface area as a whole football field! This gives them huge potential and potency compared to the bulk material.

Imagine laying out a football field with a thin layer of silver, think how much silver that would need, and then compare that with the amount that is in the spoon! This high surface area to volume ratio is one of the most important properties about nanoparticles.

With all that surface area and the energy that exists, the nanoparticles need to be held together

‘somehow’. That is where the furry parts of the tennis ball come into play. Imagine them as small molecules that hold on to the surface of the particle and stop it from breaking up under its own energy. It is like a tree whose roots can prevent soil erosion because the soil is bonded to the root in the ground. The chemical we use in this lab is mercaptosuccinic acid, and this helps to hold the nanoparticles in shape by bonding to the surface of the particles.

There are a few basic points to remember about making nanoparticles:

1) You need a nucleation point, a place for the metal (silver in this case) to start bonding to one another and start growing into a larger particle. For this you often need some ingredient that can break down a metal salt, in this case silver nitrate, which is accomplished by using sodium

borohydride. This reduces the silver nitrate into silver ions that are free then to bond with each other.

2) You need some mechanism to keep the particles at the nanoscale and stop them from ripping and growing into something much larger, this is accomplished using the capping agent mentioned earlier (mercaptosuccinic acid). A great deal of cutting edge research revolves around varying the capping agent in order to control the size of your nanoparticles and tailor them for specific tasks.

But not only can you change the size of particles in this way, you can also change the shapes.

Why silver nanoparticles?

Silver is a very easily oxidized material; it has been used already commercially for its anti-

microbial properties from athletic wear to sterilizing water. It has a very interesting interaction with light due to a dielectric constant that makes the light response occur in the visible regime.

Notably silver is one of the only metals that can be tailored to respond across the full visible spectrum.

Their light interaction can then be used in various fields such as photonics where new materials can be made to transport light in a similar fashion to the optical cables that we use now, but with a higher yield.

These waveguides act like wires and could be made smaller and lighter than present day wires, but carry more light.

Another use of this light interaction can be used for imaging in biological systems, where the nanoparticles can be used as vectors to carry drugs to specific sights because of specific capping agents being used, and the internal core can be used to image the delivery and ensure the cargo is delivered correctly to the correct location in the body. One group at Rice that is working on this exciting research is the Barron lab (<http://python.rice.edu/~arb/Barron.html>).

TA Demonstration on the Hydrophobicity of Silver

Adapted from George Lisensky's procedure that demonstrates the hydrophobicity of silver based on the Tollen's test and the ability of self-assembly of thiol monolayers (SAM) on gold surfaces: Essentially your TA will coat silver with a monolayer of octadecanethiol, effectively producing a non-polar surface and causing water that is dropped onto this surface to bead up.

Once your TA has placed a clean microscope slide in a Petri dish. Your TA will place 8 small drops (or 4 large drops) of a 0.5 M solution on the microscope slide ([Figure 12.1](#)).



Figure 12.1.

Image of a glucose solution beading on a glass slide.

Then your TA will add 25 small drops (or 12 large drops) of an [active silver ion](#) solution (made by adding concentrated ammonia drop wise to 10 mL of 0.1 M silver nitrate solution until the initial precipitate just dissolves., followed by adding 5 mL of 0.8 M KOH solution; a dark precipitate will form ([Figure 12.2](#)). Add more ammonia drop wise until the precipitate just redissolves. This "active silver" solution has to be used within an hour of preparation. CAUTION: To avoid the formation of explosive silver nitride, discard any remaining active solution by washing down the drain with plenty of water) to the glucose solution and gently agitate to mix the

solution.

Figure 12.2.

Image of the preparation of the silver layer seen as a dark precipitate.



After waiting several minutes while the solution darkens and a grayish precipitate forms, a silver mirror is also forming on the slide, though it may be obscured by the precipitate c.f., Tollen's reagent. Your TA will use water from a wash bottle to wash off the precipitate and reveal the

silver mirror ([Figure 12.3](#)) being careful to avoid contact with the solution since it will stain their hands.

Figure 12.3.

Image showing the silver mirror formed on the glass slide.

Your TA will remove the slide from the Petri dish ensuring that he/she does not touch the silver solution, and rinse the silver mirror with water. How attracted are the water drops to the surface?

(Like attracts like.) Do water drops on silver spread out or bead up?

The contact angle is between the side of a drop and the slide. Is the contact angle wide (small attraction to the slide) or narrow (large attraction to the slide)?

Your TA will wait for the surface to appear dry. (For faster drying we will use a hair dryer.) Cover the silver with a few drops of a long chain **alkanethiol solution**, octadecanethiol, in ethanol (add a small amount of octadecanethiol, to 20 mL of ethanol. When finished, dispose of this solution by adding about 5 mL of household bleach. Let stand for several minutes then wash solution down the sink).

After the ethanol has evaporated, your TA will now have an alkanethiol monolayer with the sulfur atoms bound to the silver and the hydrocarbon tails pointing away. Your TA has effectively coated the surface with hydrocarbons.

How attracted are the water drops to the surface? (Like attracts like.)

Do water drops on the monolayer coated surface spread out or bead up?

Is the contact angle greater or less than before the alkanethiol was added?

Is the water attracted more to the plain glass, to the silver, or to the alkanethiol monolayer-coated silver?

Experimental Procedure no1 - ripening of silver nanoparticle

Solutions of silver nitrate (250 mg to 500 mL) and mercaptosuccinic acid (405 mg to 500 mL) have ALL been previously prepared for you. This can be gathered from the glass bottles situated in the lab.

1. Find and open the microlab program. Ensure that the accompanying box has power and is turned on, and that it is connected to the laptop via the USB plug. Once everything is connected and you double click the microlab.exe file, a box will open in front of you.
2. In the tab labeled “New” you will find the icon for the “Spectrophotometer”, please double click this.
3. This brings up the program that we will use, at which point you should take a reading of a blank sample, this is done by filling a vial with DI water and placing in the appropriate slot, and covering with the film case. When the blank sample is in place, click the button “Read Blank”. This will generate a series of data points that you can see.
4. Please take note to read from the Absorbance tab, this is the third option on the top right.
5. In a beaker gather 50 mL of silver nitrate solution; use the photo spectrometer to take an absorbance measurement of the silver solution on its own. To do this place twenty drops (approx 1 mL) into the glass vial provided, and dilute to the top with DI water. Shake the vial twice to ensure the solution is homogenous. Record the UV-Vis spectrum.
6. Place the 50 mL silver solution into the Erlenmeyer flask and start stirring, your TA will have the stir bars.
7. Complete the same process as above but this time uses 50 mL of the solution of mercaptosuccinic acid. Please make sure to rinse out the glass vial thoroughly. Take a spectrum using the photo spectrometer of the MSA solution.
8. Now place the 50 mL of MSA solution into the Erlenmeyer with the silver that you are already stirring vigorously – this means stirring at such a rate that you can see a vortex created.
9. Take a spectrum of the solution that is currently stirring, this should include the MSA and

silver. You should notice, similar to DI water, and the MSA and silver on their own, that there is no response in absorbance of either of the chemicals when analyzed combined that would indicate that the nanoparticles have not been formed yet, but all the final ingredients are already there. This is why we need to add the sodium borohydride, as this will break the silver nitrate up so the silver can bind with the sulfur atom in the MSA molecule and the silver ions can start bunching up and the nanoparticles can begin to form.

10. Make the sodium borohydride solution by diluting 300 mg with DI water to 100 mL using a volumetric flask. From the equation of the chemical, NaBH_4 , and upon the addition of water did you notice anything occurring, what happened in the reactions? (Did it change color, consistency, did gas evolve) what could have caused this? Sodium borohydride is highly hygroscopic, that means it reacts very readily with water, and the water in the atmosphere (especially in Houston) could have caused the salt to harden – this means you may have to coax the salt a little with some prodding using the spatula – without breaking the spatula!

11. To the solution of silver nitrate and mercaptosuccinic acid that is stirring place a few drops of the sodium borohydride solution, note the color change, then place some more sodium borohydride and note the color change again. What do you think is happening here, and why is the colors changing? What were the colors of the solution? Stop adding sodium borohydride when the color has reached a steady state and is not changing anymore. If you add too much sodium borohydride the products could come out of solution.

12. When you have a steady dark, black coffee color, you have added sufficient sodium borohydride. Immediately take 20 drops of your Ag nanoparticle solution and place into the glass vial; make sure it is diluted down to a slightly sandy color by the addition of DI water.

Place the filled vial into the slot and acquire spectroscopic data in the same manner as before, this will be your “time = 0” run – now what is different this time, compared with the last spectra you acquired? And how has this occurred?

13. Continue to repeat this process of taking 20 drops of your Ag nanoparticle solution and dilute in a vial and take spectroscopic data every 5 minutes for a total of 50 minutes,

14. When you are finished completing the experimental runs, then you can export the data so that it can be graphed in Excel.

15. Go to File, then scroll down to 'Export Data as.' and then select "Comma separated values'.

This generates a file ending in .csv that can be later opened and used in Microsoft Excel;

please take note where the file was saved.

16. Use excel to graph each of the data sets on the same graph. Complete an Area graph, with 3D

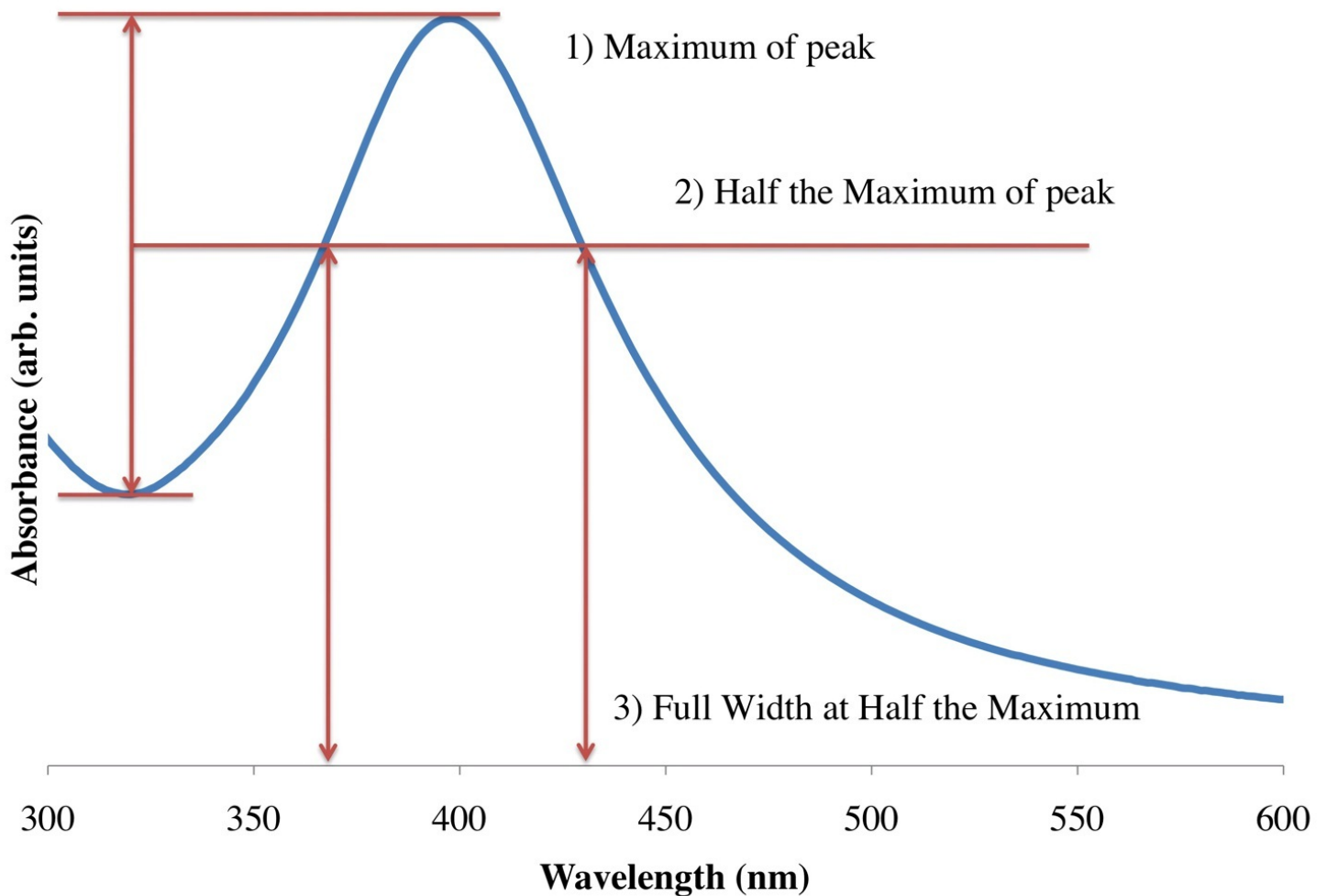
visualization to graph the ripening process. You should notice some of the intensity changing

from the first data set until the last – why do you think this is happening?

17. Using Excel again take the data from the first profile and data from the profile with the tallest peak. Graph each one separately. This is to demonstrate how the nanoparticles ripen over time.

So you can see there is some movement in the system and it can take several hours before the

entire system reaches some equilibrium. Note: You will not see the trough that we showed you



in the PowerPoint as MicroLab does not go down to 310 nm, which is why we are posting data so that you can calculate the full width half maximum..

18. You have now successfully made some silver nanoparticles! Congratulations.

19. Finally plot a graph from the data posted online in order to calculate the diameter of the nanoparticles once you have extrapolated the full width half maximum (FWHM), using the equation:

$$D = 230 / (\text{FWHM} - 50).$$

For help on getting the FWHM use [Figure 12.4](#).

Figure 12.4.

A plot demonstrating the three steps to obtain the FWHM from a graph of the UV-vis spectrum for the silver nanoparticles.

Experimental procedure no2 - lasers and colloids

When the nanoparticles form they are able to scatter huge amounts of light. As atoms the silver will not scatter any light, but when it is made into the form of nanoparticles the light scattering is possible to see using a simple laser light. Large chunks of silver cannot be soluble in water but in the nanoparticle form it can be. We can use the laser pointers to see when the nanoparticles are forming.

During the first reaction you can see how sodium citrate takes a long time to create nanoparticles, at the beginning the laser does not shine through the solution. But over a period of about 20 minutes you will be able to see the nanoparticle form by using the laser light. When the nanoparticles have formed you will be able to see the laser run through the solution.

When you make the nanoparticles with sodium borohydride now, it is so strong it will make nanoparticles much faster. This reaction only takes about 2 minutes to occur.

Aim

1. To synthesize silver nanoparticles using two different reducing agents
2. To see the difference in the rates of reaction between the two reducing agents
3. To use laser pointers to determine when the nanoparticles form and hence, to see which reducing agent works faster

Sodium Citrate

1. In a glass vessel gather 75 mL of silver nitrate solution. Put this on a hot plate and place a stir bar inside it. Start heating the solution with a medium setting, start the stir bar so that a small vortex occurs in the solution.
2. After a period of five minutes when the solution is getting warmer, place 2 mL of tri sodium citrate into the solution that is warming on the heat place. Make sure to add the sodium citrate drop wise. This will take about two minutes to do.
3. Now use a laser pointer to see if any nanoparticles have formed.
4. Over a period of 20 minutes you should notice a change in color that occurs because of the formation of the silver nanoparticles. Continuously use the laser pointer to look for the formation of nanoparticles.
5. Turn off the hot plates and take the glass away from the heat.
6. When the solution has cooled to room temperature place the waste in the waste container.

Sodium Borohydride

1. In a glass vessel gather 75 mL of silver nitrate solution. Place a stir bar inside the reaction vessel and start stirring with a speed that creates a small vortex.
2. Gather 50 mL of mercaptosuccinic acid (MSA) and place this inside the same vessel as the silver nitrate that you have recently got.
3. Use the laser to light to see if there are any nanoparticles present.
4. Now get your TA to help distribute some sodium borohydride for you. This is a very reactive chemical and will lose strength over time. Your TA will place sodium borohydride in the reaction vessel.
5. While your TA is adding the sodium borohydride, check to see if any nanoparticles are forming by using the laser light.
6. When the reaction is completed place the materials in the waste container.
6. When the reaction is completed place the materials in the waste container.
7. Clean all your glassware.

Conclusion

Nanoparticles are an exciting and emerging technology. There is much to learn about how to use these new structures. It is a delicate and complex process to learn how to make things so small, but as you have discovered today, it is not impossible to do. The detection of nanoparticles can be easily achieved with the use of a hand held laser pointer. This is due to the extremely large scattering cross section that nanoparticles have.

Additional Information

We use light to see things around us, that light has a certain size, or wavelength. And if something is smaller than light, we cannot use the light to see it directly, so we have to use things with smaller wavelengths. Let's use an example, consider a hand of a certain size, and some hieroglyphics on a wall (er?). With very large hands the details in the wall are difficult to make out, but still you can note that they are there. But when you use smaller and smaller hands the details become easier to make out. It's the same kind of idea when using the light. For us as human beings it is not usually a problem in our everyday lives, the size of the light is much smaller than the artifacts we deal with as we move around. But when looking at smaller and

smaller things as in the nanoscale, we can't use visible light because the light passes right over the objects normally, and it's as if they don't exist.

One trick around this is to use shorter wavelengths of light, like using X-rays at the hospital to image brakes and fractures of the skeletal system. And in nanotechnology what we often use are

electrons, Because the wavelength of the electrons are far smaller than the object we are looking at, we can get a good picture of what is going on at the nanoscale. There are two main instruments to do this: the TEM (transmission electron microscope), and the SEM (scanning electron

microscope). In the same way that the X-rays at the hospital pass through the skin but not the

bones, the TEM accelerates electrons through materials, and depending on the type and size of the material the electrons either pass through or not. And we get a black and white image of our

system at the nanoscale. In [Figure 12.5](#) you see a picture of the type of silver nanoparticles that you made in the lab, this was taken with a TEM in Dell Butcher Hall here at Rice. The dense silver particles don't allow the transmission of the electrons, and we get a black and white picture of the nanoparticles. This has been calibrated and can be used to tell us the size of the particles; they are around 10 nm on average.

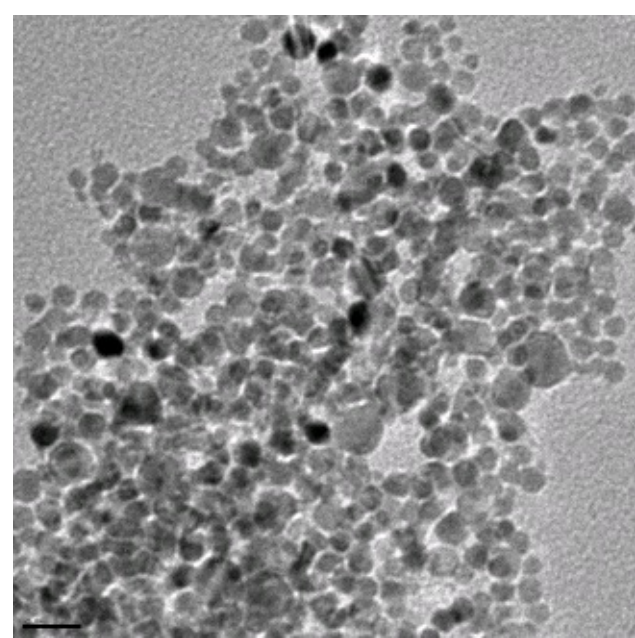


Figure 12.5.

TEM image of silver nanoparticles, scale bar is 20 nm.

But when electrons pass through the material it is not always a clean break, some of the energy

can be imparted on the materials and so it won't pass all the way through. This can cause a

secondary effect that depends on the material that is being imaged, and this is essentially how the SEM works. Instead of electrons passing through like in the X-rays in the hospital, the materials you image have a reaction to the bombardment of electrons in the electron beam. In [Figure 12.6](#)

you see a bunch of larger silver nanoparticles that have been imaged using an SEM here in Dell Butcher Hall at Rice University.

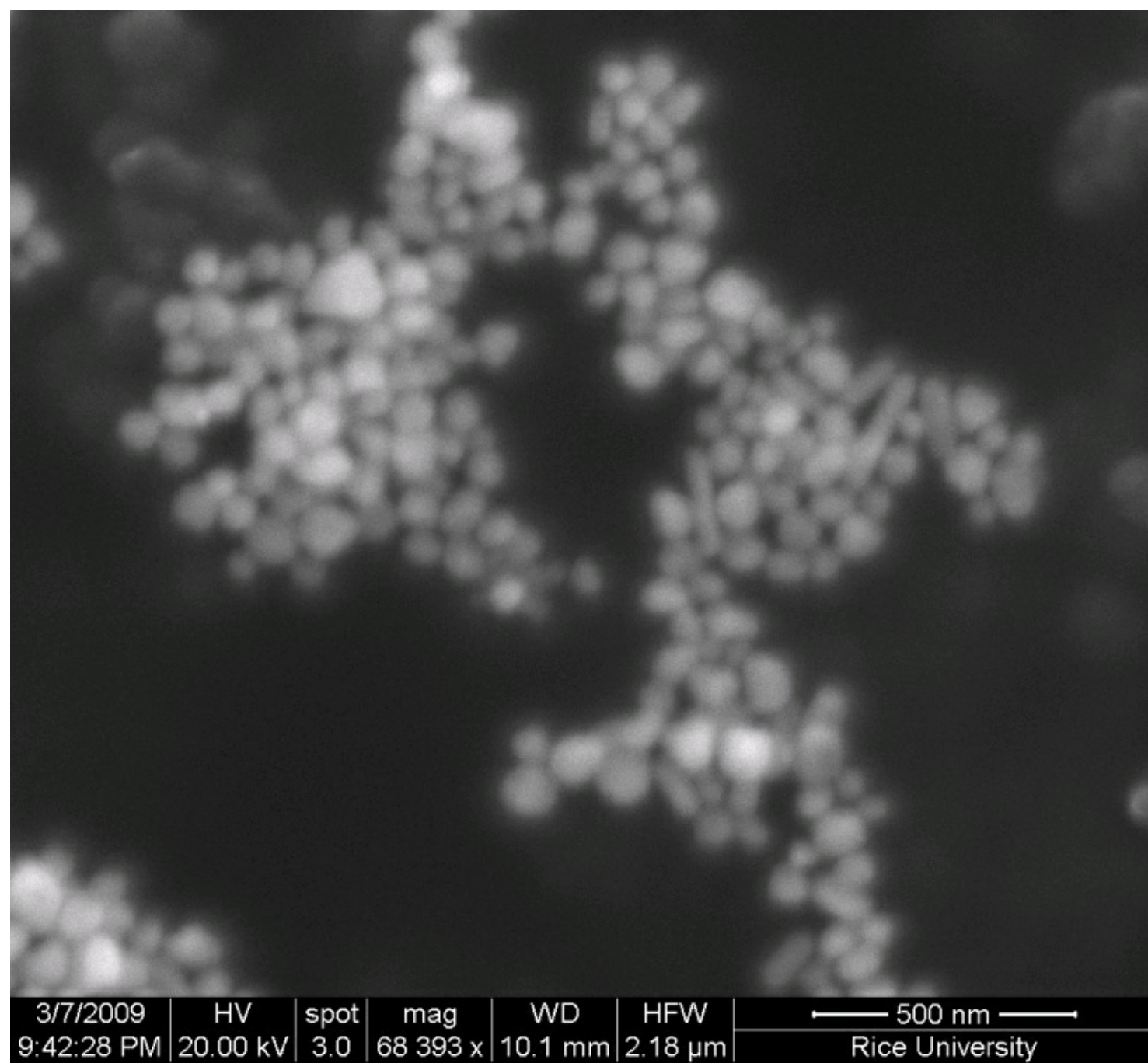


Figure 12.6.

SEM image of larger silver nanoparticles, scale bar is 500 nm.

Solutions

-
-
-
-

Chapter 13. Thermochemistry

Thermochemistry

We will begin lab in the amphitheatre of DBH

Objective

To explore heat transfer through calorimetry.

To use calorimetry to determine the enthalpy of reaction of a strong acid and a strong base.

To use Hess's Law of Heat Summation to determine the heat of hydration in calcium chloride.

To explore a common use of heat of reaction in real life.

Grading

Pre-Lab (10%)

Lab Report Form (80%)

TA Points (10%)

Background Information

When two objects at different temperatures are brought into physical contact, thermal energy will spontaneously transfer from the warmer object to the colder object until both objects have

achieved the same temperature. Assuming the two objects are thermally insulated from their

surroundings, the heat lost by the warm object is identical to the heat gained by the cold object.

This is a manifestation of the Law of Conservation of Energy.

The heat transfer, q , is a function of the mass of the object (m), the change in temperature

undergone by the object (ΔT) and the object's specific heat (C_s). This statement can be expressed mathematically as

$q =$

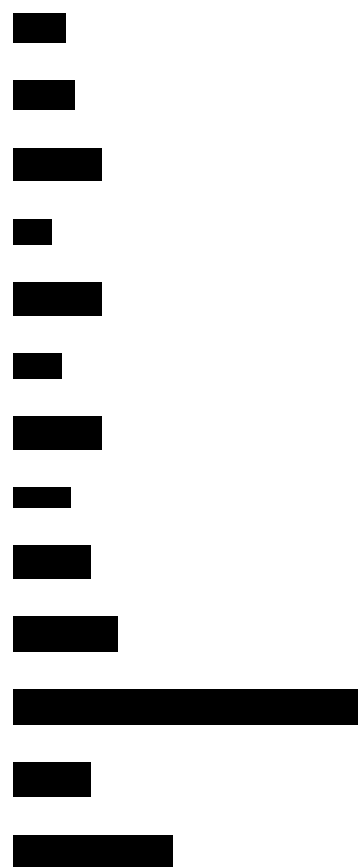
Temperature change is always defined as

-

, which means that q for the hotter object (

) is negative and q for the colder object (

) is positive. If energy is conserved, then


$$\begin{aligned} & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \\ & \blacksquare \end{aligned}$$

+

= 0

and

(

) + (

)

= 0

Now consider dropping an ice cube into water just warm enough to melt the ice cube but not warm enough to further heat the water from the cube. The observation is that the ice cube melts and the warm water cools to 0°C . It is important to recognize that during the phase change, the temperature of the ice cube does not change. Therefore, it is not possible to use the preceding

equation to determine the heat transferred. Rather, the energy transferred to the ice cube from the warm water affects the phase change. The energy equation is now adjusted to incorporate the

enthalpy required to melt the ice cube, ΔH_f (where f stands for fusion):

(
)

$$+ \Delta H_f = 0$$

It is also possible to have thermal energy when chemical reactions occur. The amount and direction of heat flow is dependant on the chemicals reacting. Using a calorimeter, it is possible to experimentally determine the heat of reaction.

Calorimetry

In the technique known as constant-pressure calorimetry, enthalpies of phase changes or chemical reactions are determined indirectly by measuring temperature (at constant pressure) changes in a medium, most often water, surrounding the materials undergoing the change. That is, by

measuring ΔT of the water one can use the preceding equation to calculate ΔH for the process of interest. Of course, this means one must know the mass of the water used and water's specific heat:

$$= 4.18 \text{ J/(gK)}.$$

Today in Part I, you will add a strong base to a strong acid, measure the temperature change in the water as the two react, and use that information to calculate the heat of reaction per gram of

NaOH. Then convert your experimental value into an enthalpy in kJ/mol (of NaOH).

The enthalpy of a reaction might be difficult to obtain directly by experiment but can be determined by measuring (or looking up in tables) the enthalpy changes of reactions which contain the reactants and products in a process governed by Hess's Law of Heat Summation. In

Part II of this experiment you are asked to find the enthalpy of hydration of

the "target

reaction" in this manner.

Target reaction:

It cannot be measured directly because of the slow kinetics of the reaction in the solid state.

However, the heats of dissolution of

and

can be determined and, using Hess's

Law, the enthalpy of hydration can be calculated.

Theoretical value of the enthalpy of hydration of

anhydrous is -81.33kJ/mol . Theoretical

value of the enthalpy of hydration of

is 15.79 kJ/mol .

Experimental Procedure

Materials Required

two Styrofoam cups (serve as the calorimeter)

styrofoam cover (top to the calorimeter)

conventional or digital thermometer

stirring motor and stir bar

two large Ziploc bag

two plastic spoons

Setup of the MicroLab Thermistor

Open the MicroLab Program by clicking on the Shortcut to MicroLab.exe tab on the desktop.

On the “Choose an Experiment Type” Tab, enter a name for the experiment, and then double

click on the MicroLab Experiment icon

Click “Add Sensor”, Choose sensor = Temperature (thermistor)

Choose an input, click on the red box that corresponds to the port that your thermistor is connected to.

Label = Thermistor, sensor units = $^{\circ}\text{C}$, click next

Click “Perform New Calibration”

Click “Add Calibration Point” place the thermistor and thermometer in an ice water bath. Wait

until the temperature is constant and then read the temperature on the thermometer (to the

nearest 0.1 °C) and enter it into the “Actual Value” box in MicroLab and hit “ok”.

Again, Click “Add Calibration Point” place the thermistor and thermometer in warm water bath. Wait until the temperature is constant and then read the temperature on the thermometer (to the nearest 0.1 °C) and enter it into the “Actual Value” box in MicroLab and hit “ok”.

Under Curve Fit Choices , click on “First order (linear)” and then “Accept and Save this Calibration”, when prompted to enter units, set as deg C. Save as your name-experiment-date.

Click “Add Sensor”, Choose sensor = Time

Choose an input, click on the red box that corresponds any of the Timers.

Label = Time 1, click next, click Finish.

Left click on thermistor and drag to the Y-axis over “data source two”, also click and drag to column B on the spreadsheet and also click and drag to the digital display window.

Left click on Time and drag to the X-axis over “data source one”, also click and drag to column A on the spreadsheet and also click and drag to the digital display window.

When you are ready to obtain data, click start. When you are finished collecting data, click stop. To run another trial, click repeat experiment.

Part I. Reaction of Strong Acid with Strong Base

1. Weigh the calorimeter (styrofoam cups with lid) WITH a stir bar. Accurately measure 50 mL of 1 M HCl into your calorimeter. Turn on the stirring motor to the medium speed. Fit the thermistor through the cardboard lid to a length such that its tip goes deep into the solution but misses the stir bar. Cover the calorimeter.
2. Start the Acquisition program. You should see successive, constant temperature readings of near room temperature. Accurately measure 50 mL of 1 M NaOH and quickly add it to the acid solution in the Styrofoam cups. Cover the calorimeter.
3. Continue monitoring the temperature change until thermal equilibrium is reached (the temperature stops changing or starts decreasing).

4. Stop the Acquisition program. SAVE YOUR FILE. Remove the cardboard lid, reweigh the calorimeter and record the information on your lab report.

5. Repeat the same procedure (Steps 1-4) two more times. Save all your files. Give them different, distinguishable names.

Print off your graphs and use the data to determine the initial and final temperatures. Record all the temperatures to the nearest 0.1 °C. If a slight downward trend appears on the final temperature plateau, use the maximum value achieved. Calculate the average enthalpy of the reaction and

standard deviation for the three trials. Then combine your data with the data obtained from the

rest of the group and calculate average enthalpy of the reaction and standard deviation for all trials together. Compare your average and standard deviation with that of the larger set and comment on the results obtained using a larger data set.

Part II. Enthalpy of Reaction

http://www.ajdesigner.com/phpstatistics/standard_deviation_sample.php

The same basic procedure you used in the dry ice sublimation lab will be used here today.

1. Tare your calorimeter and pour approximately 75 mL of cold water in it. Add stir bar. Record the mass to 3 decimal places. Place the cups on the magnetic stirrer and turn on the stirring

motor to a medium rate. Make sure you can fit the thermistor through the styrofoam cover to a

length such that its tip goes deep into the water but misses the stir bar but do not insert it.

Cover the calorimeter.

2. Start recording data on the MicroLab interface. Let it run for about 10 seconds before putting thermistor through the Styrofoam cover. You should see successive, constant temperature

readings of near room temperature. Approximately 5 grams of anhydrous has been weighed for

you, record the weight of this plus the weight of the weighing bottle. Then weigh the dry

empty weighing bottle. Quickly add to the water and reposition the thermistor and cover

assembly.

Note: Since anhydrous absorbs moisture rapidly from the air, close the lids of the bottles securely immediately after using. Dry the spatula each time before weighing the powder and clean the

balance of any solid.

1. Continue monitoring the temperature change until thermal equilibrium is reached (the

temperature stops changing or starts decreasing).

2. Stop collecting and SAVE YOUR DATA and/or print it.

3. Repeat steps 1-4 two more times. Don't forget to save your data each time you do a run because it is lost as soon as the next run begins.

4. Repeat the process but replace anhydrous with .

5. Use the data to determine the initial and final temperatures. Record all temperatures to the nearest 0.1 . If a slight downward trend appears on the final temperature plateau, use the

maximum value achieved. Calculate the average enthalpy of dissolution and standard

deviation. Use Hess's Law to calculate the heat of hydration of and % error. Then combine

your data with the data obtained from the rest of the class and calculate the average enthalpy

of dissolution and hydration.

Calculations

Calculations are similar to those done for the acid-base neutralization reaction. The calculation of is the same as the calculation of , that is:

=

q_{water} can be calculated using water's specific heat, mass of water and temperature change of

water solution:

In order to account for the mass of anhydrous or hexahydrate, divide by the mass of anhydrous or to get in J/g. Then convert to J/mol.

Part III. Chemistry of Life

Hot packs and cold packs are a real life example of thermochemistry. Anhydrous magnesium

sulfate and ammonium chloride can be used to make "hot/cold" packs similar to those used for

sports injuries and in hospitals. Your TA will make a pack from each of the two compounds and

pass them around and answer some fundamental thermochemical questions about the reactions

involved.

TA Procedure

Fill two Ziploc bags half way full with water.

Put two spoons full of anhydrous magnesium sulfate into one bag and two spoons full of ammonium chloride into the other bag, zip them closed, and shake.

Pass the packs around to students and observe the temperature change.

Solutions

Index

Attributions

Collection: **Gen Chem Lab**

Edited by: Mary McHale

URL: <http://cnx.org/content/col10452/1.51/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Avogadro and All That**

By: Mary McHale

URL: <http://cnx.org/content/m31916/1.8/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/3.0/>

Module: **Stoichiometry: Laws to Moles to Molarity**

By: Mary McHale

URL: <http://cnx.org/content/m15086/1.14/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Totally, Terrific Table**

By: Mary McHale

URL: <http://cnx.org/content/m17556/1.6/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Going Green With AI**

By: Mary McHale

URL: <http://cnx.org/content/m32209/1.7/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/3.0/>

Module: **VSEPR: Molecular Shapes and Isomerism**

By: Mary McHale

URL: <http://cnx.org/content/m15100/1.8/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Nanotechnology: Ferrofluids and Liquid Crystals**

By: Mary McHale

URL: <http://cnx.org/content/m15532/1.10/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Solid State and Superconductors**

By: Mary McHale

URL: <http://cnx.org/content/m15259/1.8/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Colorful Copper**

By: Mary McHale

URL: <http://cnx.org/content/m15103/1.14/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Metathesis: To Exchange or Not?**

By: Mary McHale

URL: <http://cnx.org/content/m15130/1.6/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Electrochemistry/Alchemy: Molar Mass of Cu and Turning Cu into Au**

By: Mary McHale

URL: <http://cnx.org/content/m15189/1.3/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Bonding 07**

By: Mary McHale

URL: <http://cnx.org/content/m15205/1.4/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

Module: **Silver Nanoparticles: A Case Study in Cutting Edge Research**

By: Alvin Orbaek, Mary McHale, and Andrew Barron

URL: <http://cnx.org/content/m19597/1.11/>

Copyright: Alvin Orbaek, Mary McHale, and Andrew Barron

License: <http://creativecommons.org/licenses/by/3.0/>

Module: **Thermochemistry**

By: Mary McHale

URL: <http://cnx.org/content/m15264/1.7/>

Copyright: Mary McHale

License: <http://creativecommons.org/licenses/by/2.0/>

About Connexions

Since 1999, Connexions has been pioneering a global system where anyone can create course materials and make them fully accessible and easily reusable free of charge. We are a Web-based authoring, teaching and learning environment open to anyone interested in education, including students, teachers, professors and lifelong learners. We connect ideas and facilitate educational communities. Connexions's modular, interactive courses are in use worldwide by universities, community colleges, K-12 schools, distance learners, and lifelong learners. Connexions materials are in many languages, including English, Spanish, Chinese, Japanese, Italian, Vietnamese, French, Portuguese, and Thai.

Document Outline

- [Gen Chem Lab](#)
 - [1. Avogadro and All That](#)
 - [2. Stoichiometry: Laws to Moles to Molarity](#)
 - [3. Totally, Terrific Table](#)
 - [4. Going Green With Al](#)
 - [5. VSEPR: Molecular Shapes and Isomerism](#)
 - [6. Nanotechnology: Ferrofluids and Liquid Crystals](#)
 - [7. Solid State and Superconductors](#)
 - [8. Colorful Copper](#)
 - [9. Metathesis: To Exchange or Not?](#)
 - [10. Electrochemistry/Alchemy: Molar Mass of Cu and Turning Cu into Au](#)
 - [11. Bonding 07](#)
 - [12. Silver Nanoparticles: A Case Study in Cutting Edge Research](#)
 - [13. Thermochemistry](#)
 - [Index](#)
 - [Attributions](#)
 - [About Connexions](#)