Komar University of Science and Technology (KUST)

Fall 2016

ANALYTICAL CHEMISTRY CHM 2411L LABORATORY MANUAL

PREPARED BY
MARIAM MERRY, M.S.

Preface and Acknowledgments

Analytical chemistry is the branch of chemistry dealing with measurement, both qualitative and quantitative. This discipline is also concerned with the chemical composition of samples. It's also concerned with developing the tools used to examine these properties. Thus, it is important that students of chemistry do experiments in the Lab to more fully understand the theories they study in their textbook. Whenever it is needed, the students need to refer to the textbook, Modern Analytical Chemistry, 1st Edition. David Harvey. McGraw-Hill Higher Education 2000. The sequence of experiments in this Lab Manual is designed to follow the lecture syllabus.

It is important that you carefully prepared for each experiment by reading the related text material before coming to the lab. This way you can maximize the laboratory experience.

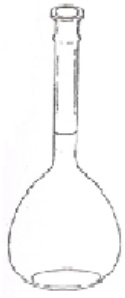
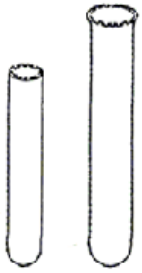
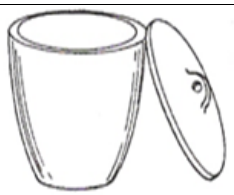
I encourage you to discuss ideas for improvements or suggestions for new experiments with me. Finally, I hope you find this laboratory manual helpful in your study of analytical chemistry for this fall semester 2016.

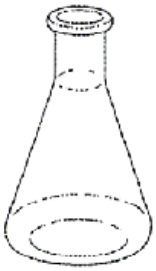
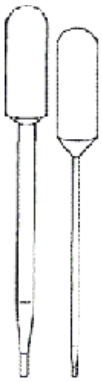
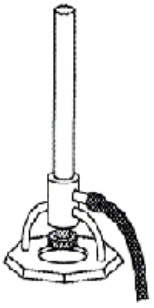
This manual was completed with support of KUST administration, and staff members. Therefore, I am thankful for their efforts.

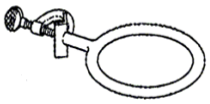
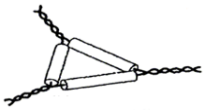
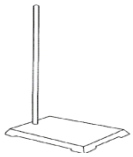
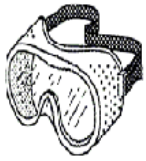
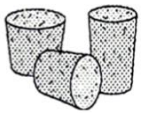
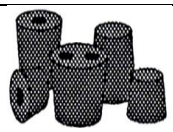
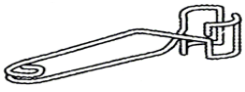
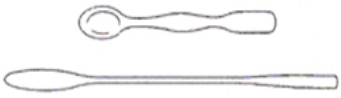
Table of Content

Common Laboratory Apparatus*

Laboratory apparatus and equipment like electronic scales, glassware, burners, graduated cylinders, and more, help the scientist conduct experiments, observe data, heat liquids, make measurements, and more.

Items Name	Picture	Items Name	Picture
Volumetric Flask		Test Tubes	
Watch Glass		Crucible and lid	
Dropper		Evaporating dish	
Funnel		Graduated cylinder	

Items Name	Picture	Items Name	Picture
Mortar and pestle		Beaker	
Stirring rod		Erlenmeyer Flask	
Micropipette		Burette	
Thermometer		Bunsen burner	

Items Name	Picture	Items Name	Picture
Wire gauze		Clamp	
Iron ring		Clay triangle	
Stand		Tong	
Wash bottle		Safety goggles	
Corks		Rubber Stopper	
Brush		Forceps	
Test tube holder		Spatulas	

Items Name	Picture	Items Name	Picture
Centrifuge		Oven	
Water bath		Electronic Lab Scale	
Hot plate		pH-meter	

* Matin J., Martin C. (2012). Catalyst: the Pearson Custom Library for Chemistry: Laboratory Experiments, Pearson Prentice Hall. ISBN13: 978-0-536-93404-8

Chemistry Lab Safety

The chemistry laboratory can be a place of discovery and learning. However, by the very nature of laboratory work, it can be a place of danger if proper common-sense precautions aren't taken. While every effort has been made to eliminate the use of explosive, highly toxic, and carcinogenic substances from the experiments which you will perform, there is a certain unavoidable hazard associated with the use of a variety of chemicals and glassware. You are expected to learn and adhere to the following general safety guidelines to ensure a safe laboratory environment for both yourself and the people you may be working near. **Each student will have to pass a lab safety exam before conducting any lab work to evaluate his/her understanding to the lab rules and safety precautions. Students who fail in that exam will have to take orientation session about lab safety before they can start lab work.** Additional safety precautions will be announced in class prior to experiments where a potential danger exists. **Students who fail to follow all the safety rules may be asked to leave the lab or suffer grading penalties.**

Laboratory Attire

1. Safety goggles *must be worn at all times* while in the laboratory. This rule must be followed whether you are actually working on an experiment or simply writing in your lab notebook. You must wear safety goggles provided by the chemistry department.

2. Contact lenses are not allowed. Even when worn under safety goggles, various fumes may accumulate under the lens and cause serious injuries or blindness.

3. Closed toe shoes and long pants must be worn in the lab. Sandals and shorts are not allowed.

4. Long hair must be tied back.

Laboratory Conduct

1. Eating, drinking, and smoking are strictly prohibited in the laboratory.

2. No unauthorized experiments are to be performed. If you are curious about trying a procedure not covered in the experimental procedure, consult with your laboratory instructor.

3. Coats, backpacks, etc., should not be left on the lab benches and stools. Beware that lab chemicals can destroy personal possessions.

4. Always wash your hands before leaving lab.

5. Be especially careful of spills around the balance. These electronic devices are *extremely* sensitive to corrosion. A brush is kept near the balance so you can brush the balance thoroughly after *each* use.

6. Clean any spill near the balance *immediately* and report it to instructor.

7. Notify the instructor immediately in case of an accident.

8. Consider *all* chemicals to be hazardous as a result never taste anything, never directly smell the source of any vapor or gases, by means of your couple hands, bring a small sample to your nose, Fig 2.2.1

Fig 2.2.1 Waft Toward Your Nose

9. Know what chemicals you are using. Carefully read the label *twice* before taking anything from a bottle.

10. Excess reagents are **never** to be returned to stock bottles. If you take too much, dispose of the excess.

11. Many common reagents, for example, alcohols and acetone, are highly flammable. *Do not use them anywhere near open flames.*

12. Always pour acids into water. If you pour water into acid, the heat of reaction will cause the water to explode into steam, sometimes violently, and the acid will splatter.

13. If chemicals come into contact with your skin or eyes, *flush immediately* with copious amounts of water and consult with your instructor.

Fig. 2.2.2 Pointing a Test Tube at Your Neighbor

14. Never point a test tube or any vessel that you are heating at

yourself or your neighbor, Fig. 2.2.2.

15. Dispose of chemicals properly. Waste containers will be provided and their use will be explained by your TA. Unless you are explicitly told otherwise, assume that only water may be put in the lab sinks.

16. Clean up all broken glassware immediately and dispose of the broken glass properly.

17. Never leave burners unattended. Turn them off whenever you leave your workstation. Be sure that the gas is shut off at the bench rack when you leave the lab.

18. Beware of hot glass--it looks exactly like cold glass.

Fire Case

1. In the event of fire, *do not panic*.

2. If a small portion of your clothes catches fire, the fire may be extinguished by patting it out.

3. *Never* use a fire extinguisher on a person. Carbon dioxide fire extinguisher is extremely cold and may cause shock to the person, frostbites, or harm to the eyes.

4. If a fire should occur in a breaker or some other container, cover it with a glass dish or other flame-retardant item.

Writing Lab Reports

The lab report for this course will be represented by a designed report sheet prepared by the course instructor and accompanying with each experiment. This sheet will illustrate the understanding of the experiment via answering some questions related to the work and it also summarize the data via tables with simple calculations whenever it is needed and simple discussion .

Submission of Lab Reports

Each group should submitted one report and the lab report of previous week experiment should be submitted at the beginning of the next experiment lab work. Any delay in submission is not acceptable unless with reasonable cause. Late submission will cause deduction from your grade one mark for each one hour lateness. No lab reports will be collected after the submitting day.

Laboratory Experiments

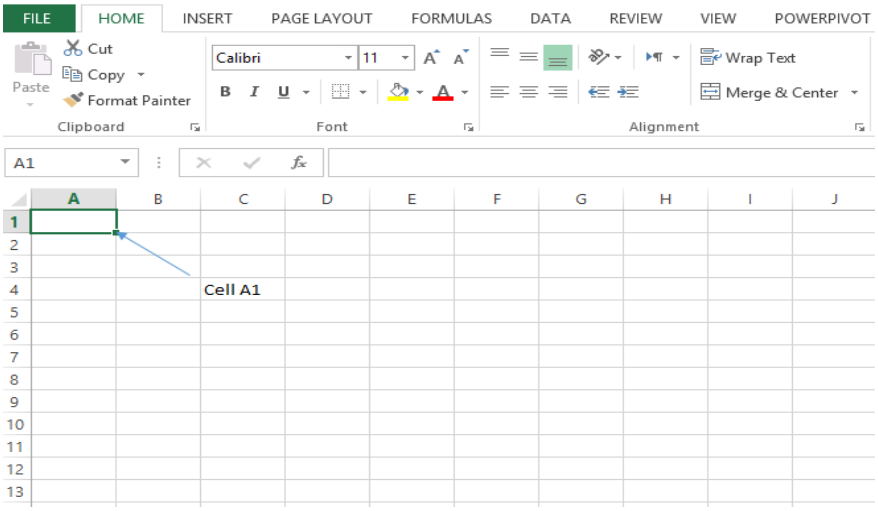
Experiment 1: Excel Tutorial and Statistical Application

Objectives

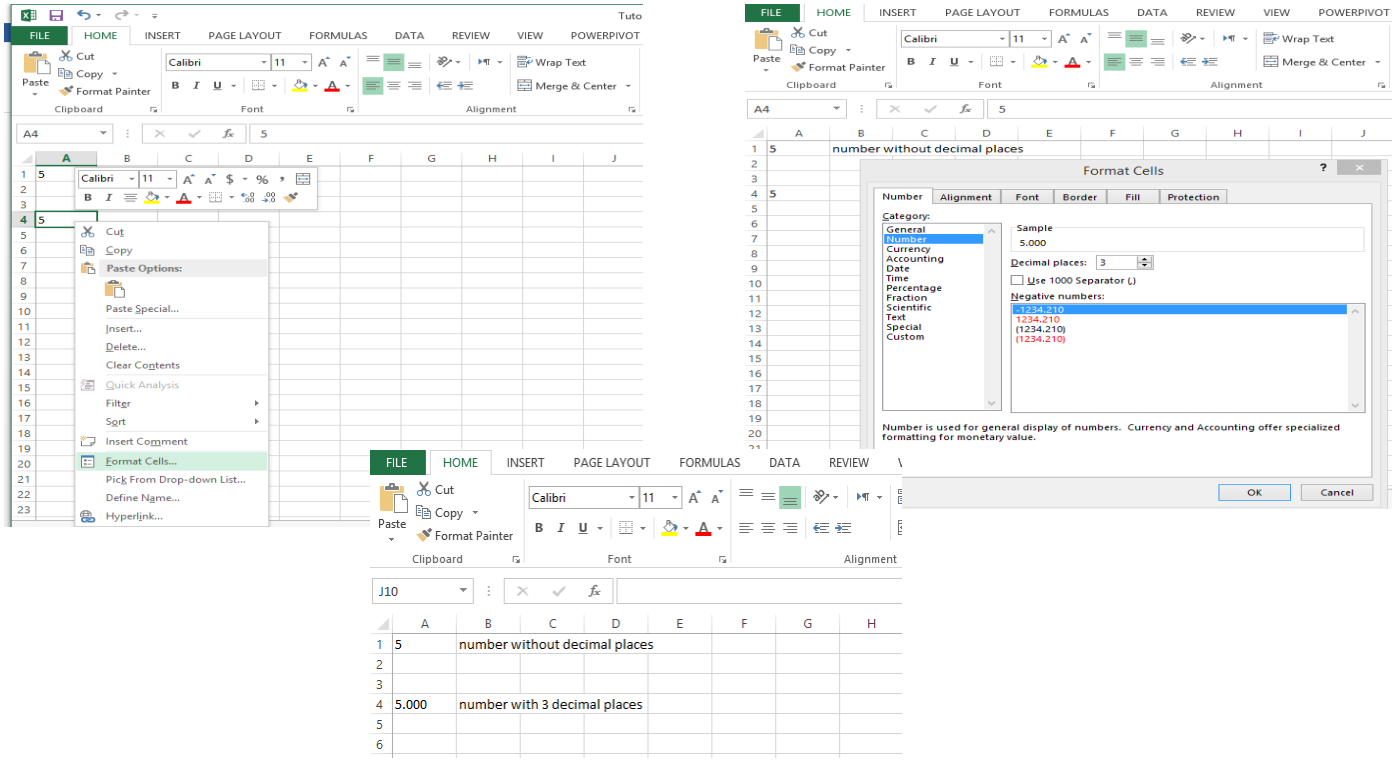
1. A brief summary of how to use Excel to calculate some simple algebraic formulas and common statistical values, and to create graphs from typical experimental data.
2. Statistical analysis express the reliability of the data being presented, it can be considered as exact treatment of uncertainty.

Introduction

When you open Excel you will see a spreadsheet with alphabetically labeled columns and numerically labeled rows. This means that each cell in the spreadsheet has a unique alphanumeric label call "cell".



You can enter data into each cell, in either alphabetic or numeric form, by simply typing whatever you need into each cell. You can move from cell to cell using the Enter key (down one cell) or Tab (across one cell) after you type in your entry. The number of decimal places displayed in the data can be changed by highlighting the data, selecting "cells" under the format menu, and choosing the "number" tab and you will see display that allows you to select the decimal places. This is especially useful for displaying calculation results with the correct number of significant figures.

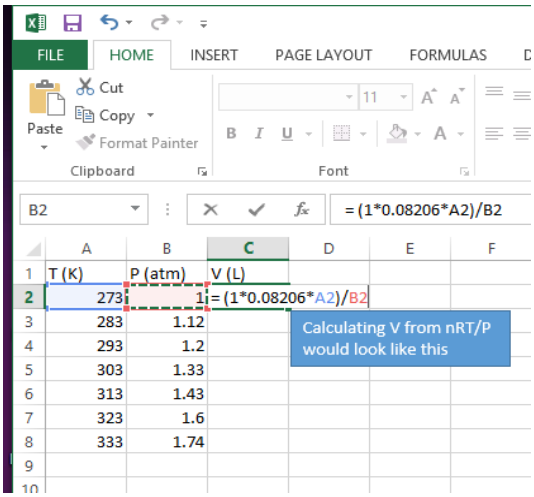


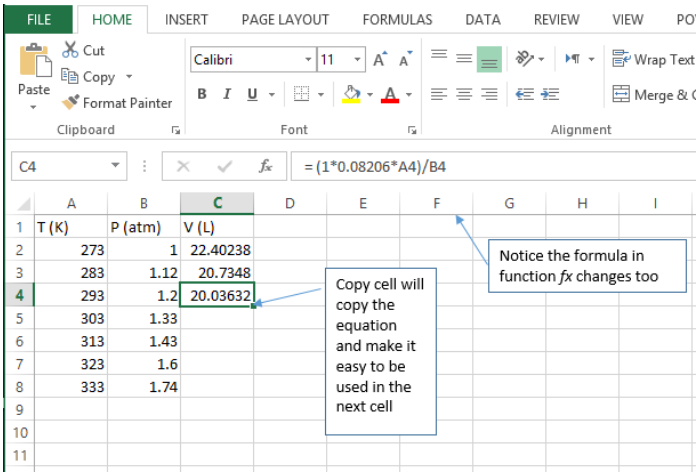
Basic Math:

Once you have your data in the columns, you need to be able to use simple expressions to manipulate the data. A mathmatical equation in Excel is always preceded by an equal sign "=".

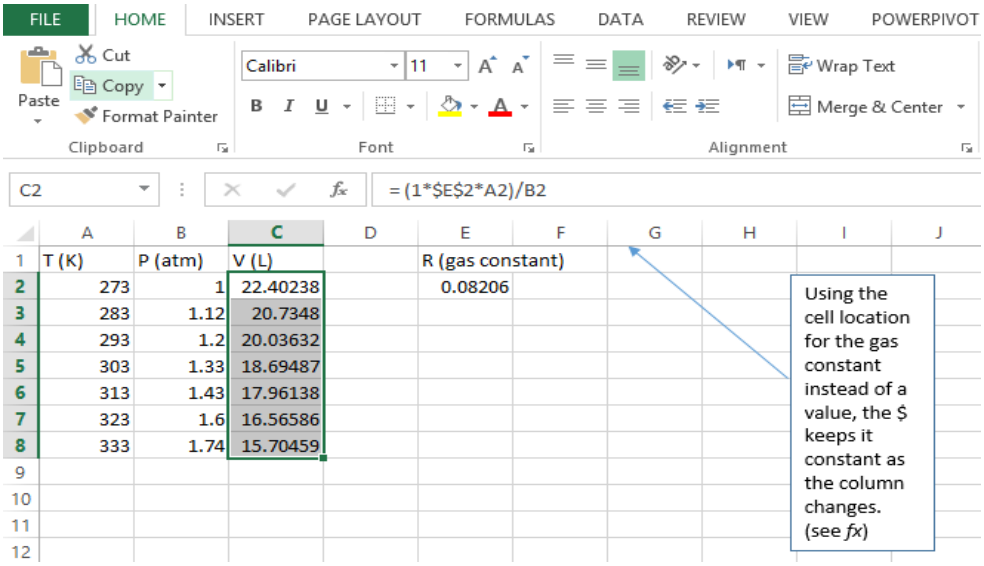
Combinations of mathmatical operations and more complex expressions cab be entered using prantheses between operations. For example:

$V = \frac{nRT}{P} = \frac{1(0.08206\frac{L.atm}{mol.K})(273K)}{1\ atm}$ = 22.4L, would be calculated as shown below:





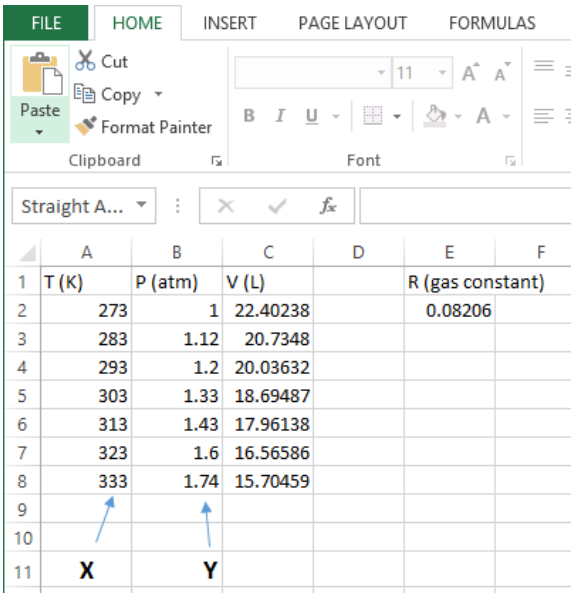
If you want a value to remain constant and not change as you copy values down a column, this can be done by simply placing a $ in front of the number and letter of the cell containing the value you do not want to change. For example, if cell E2 contains a constant in your formula, enter it as E2.



Graphing:

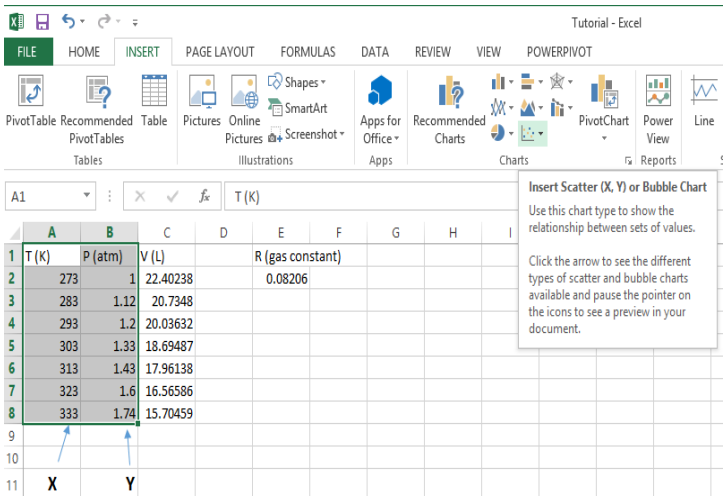
General information

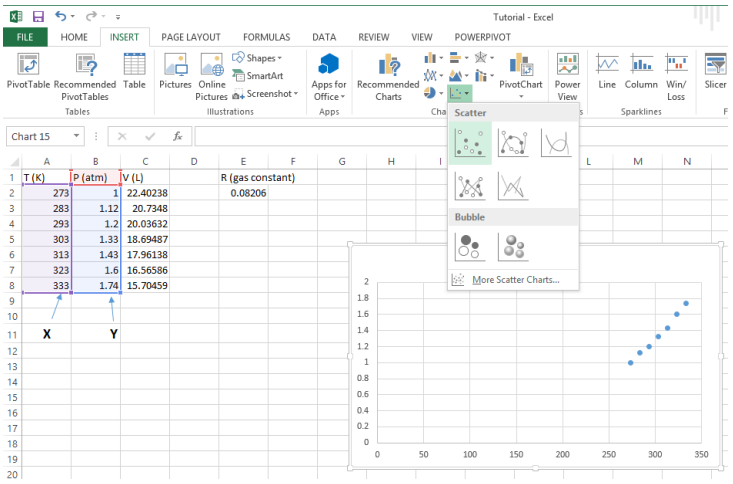
In order to create a graph in Excel, first input the data you want to graph with the x values in the column on the left and the y values in the column to its right.

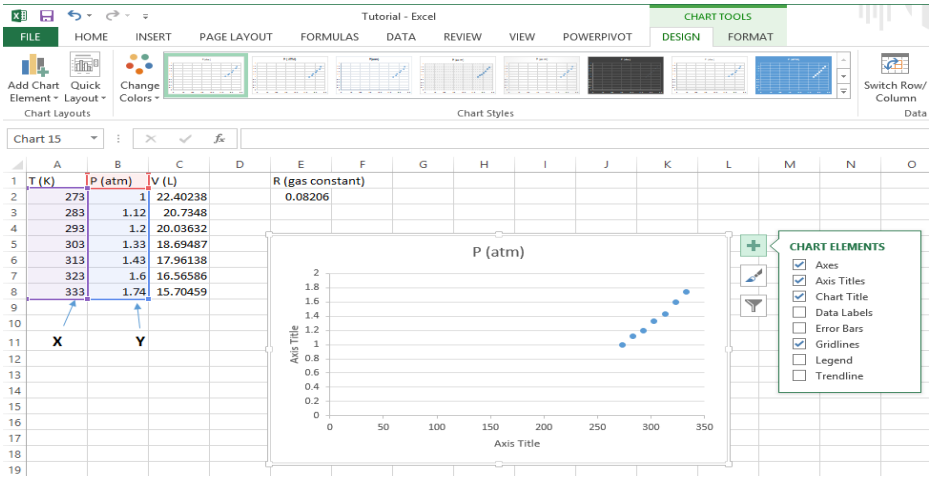


	A	B	C	D	E	F
1	T (K)	P (atm)	V (L)		R (gas constant)	
2	273	1	22.40238		0.08206	
3	283	1.12	20.7348			
4	293	1.2	20.03632			
5	303	1.33	18.69487			
6	313	1.43	17.96138			
7	323	1.6	16.56586			
8	333	1.74	15.70459			
9						
10						
11	X	Y				

Highlight the columns you wish to graph, use the insert from the toolbar and select charts, then select the type of graph you want to make. Most graphs for laboratories will be XY scatter plots.

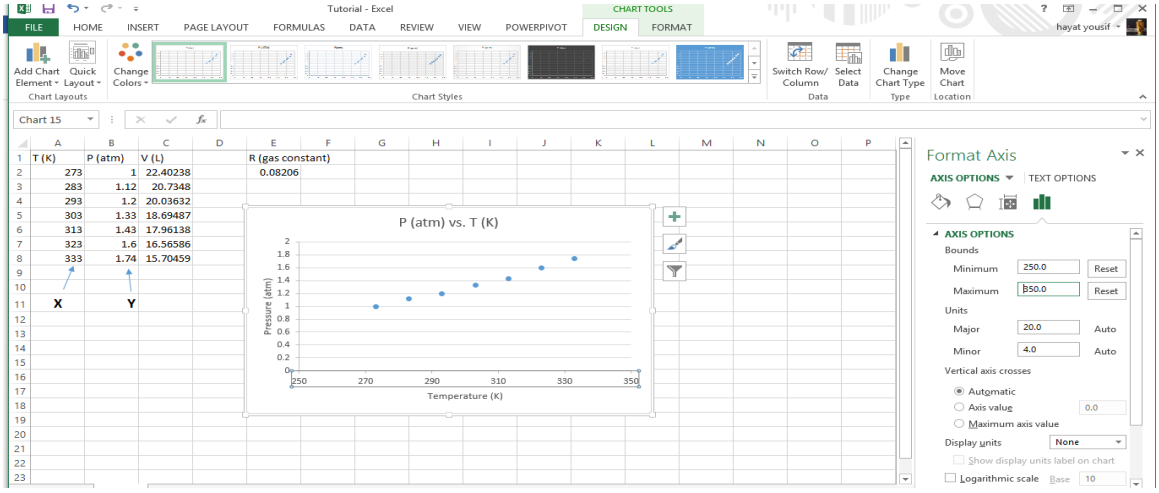




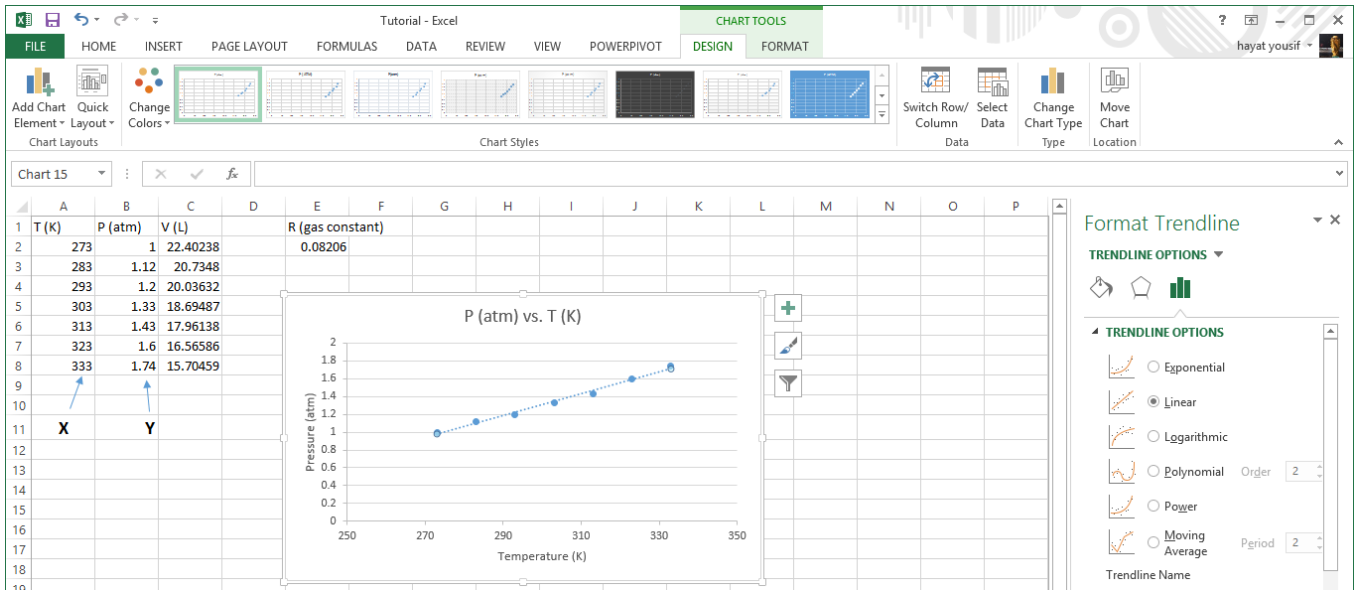


Once you select the graph, you can add some parameters like a title for X and Y axis, gridlines and the overall title for the graph by pressing on the (+) sign beside the graph:

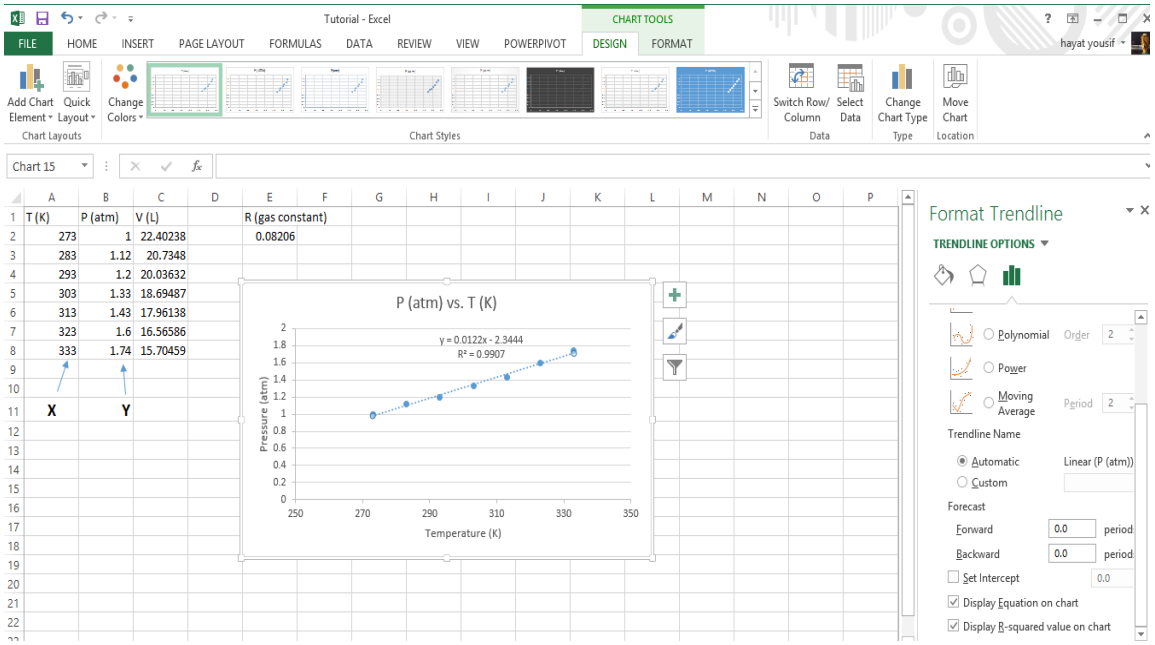
To adjust the scale so that your data points fill the graph completely, double-click on the axis you want to adjust and change the maximum or minimum values as needed.



In order to add a trend line, right click on one of the data points in the line and select from the box "Add trendline", then select the type of your line.



You can also choose to display the slope and equation of the line to your graph. The R-squared value is a statistical measure of how well your data fits the y = mx + b equation. A value very close to (1) indicates a good fit. This value is often displayed to verify the *credibility* of the data and the graph. This graph is called ***The Calbration Graph*** and it is one of the common type of graphs used for data analysis. It is a graphic representation of standard reference data used to calibrate some variable in an unknown sample.



Statistics

Statistics are away to easily express the reliability of the data. In order to express data in a manar that can easily be critiqued , there are several statistical methods must be mastered which are the analysis of errors in repeated measurements and the analysis distributions and central tendencies.

Error Analysis:

Measuring the same thing over and over again often ends up with slightly different results no matter how hard you try. This is solely due to the fact that every measurement has some error or uncertainty associated with it. Even advance instrumentation such as radar guns have errors associated with them

a. *The mean:*
   Finding the mean will help to report the data correctly. It is found by adding each individual data (x_i) and devide by the total number of measurements (N):

$$\text{Mean } (\bar{X}) = \frac{\sum_i xi}{N}$$

b. *Standard Deviation:*
   It measures how closely data are clustered about the mean value:

$$\text{SD } (\sigma): \sqrt{\frac{\sum(x-xi)2}{N-1}}$$

   In general, the smaller the standard deviatiton, the closer the mean wll be to the true value. The numerator in the equation calculates how much each individual measurment differs from the mean. The square and the square root, they take care of the fact that some data are larger than the mean while others are smaller than the mean.

Note: some statistacal approaches require the ***variance*** which is the *square of the standard deviation.*

c. *Confidence (μ) and the student's t:*

This statistical value incorporates several other values including the mean, standard deviation in the mean, and the student's t. the value is calculated by taking the mean and adding the corresponding confidence interval. (t) is the value of the student's t at a given number of degrees of freedom and confidence:

$$\text{Confidence } (\mu) = \bar{X} \pm \frac{t \,.\, \sigma}{\sqrt{N}}$$

The value t is vital, as scientifc data generally expressed at 95% and 99% confidence. Using the table below, we can select the t value based on the fact that we have (N-1) degree of freedom.

Degree of Freedom	90%	95%	99%
1	6.31	12.70	63.70
2	2.92	4.30	9.92
3	2.35	3.18	5.84
4	2.13	2.78	4.60
5	2.02	2.57	4.03
6	1.94	2.45	3.71
7	1.90	2.36	3.50
8	1.86	2.31	3.36
9	1.83	2.26	3.25
10	1.81	2.23	3.17
11	1.80	2.20	3.11
12	1.78	2.18	3.06
13	1.77	2.16	3.01
14	1.76	2.14	2.98
Infinite	1.64	1.96	2.58

This table taken from Analytical Chemistry, An Introduction, 7th Edition: Table 7 – 2 p. 152

d. *Q-test*:

It is a method dedicate entirely to determine if one particular data point (outlier point) can be rejected from the others.

$$Q_{\text{calculated}} = \frac{Xa - Xb}{Range}$$

Qcalculated is the difference between the questionable result (outlier point) and its nearest neighbor is divided by the range of the values (diffrence between the bigger and smaller value). This calculation should be done by first arranging the data in an ascending or decending order. If the value of Q calculated is found to be greater than Q table, then the data can be rejected.

Q Table (90% Confidence)	Number of Obs. (N)
0.76	4
0.64	5
0.56	6
0.51	7
0.47	8
0.44	9
0.41	10

This table taken from Quantitative Chemical Analysis, 5th Edition: Table 4 – 5 p. 82

Pre-lab Questions

1. You are asked to calibrate 10.0 mL volumetreic pipette by weighing the mass of water delivered by the pipette. You weigh six samples of water by the pipette and convert the mass of each volume by dividing by the denity of water 25°C (0.997048 g/mL). Following are your measurements:

   Calculate the following statistical measures for this data: Mean, standard deviation, variance, and the confidence interval of 90%, 95%, 99% = 9.9958 ± ---------- .

9.9690 mL	9.9700 mL	10.0360 mL	9.9650 mL	10.0150 mL	10.0200 mL

2. The table below contains data that were collected to create a calibration curve in which the absorbance of a substance of a substance is ploted versus samples of known concentration.

Known concentration (M)	Absorbance (AU)
0.10	0.115
0.20	0.260
0.30	0.485
0.40	0.790
0.50	1.175
0.60	1.640

   Graph these data and determine from the graph the concentration of the following unknown solutions:
   Solution A: 0.163 AU:
   Solution B: 0.658 AU:

3. The table below shows some data in which the peak height of triplicate sample of a substance is listed as a function of the quantitiy of material injected.
   a. Using Excel sheet, graph the average peak height versus sample mass. Apply a best-fit line to the resulting graph.
   b. Again using the spread sheet program, calculate: sample mean, variance, standard deviation and confidence intervals 95% for the data.

Gas Chromatography Calibration			
Sample mass (µg)	Peak height (cm)		
	Trial 1	Trial 2	Trial 3
10	2.6	2.3	2.4
20	3.8	4.2	4.8
30	6.2	6.5	5.9
40	9.0	8.7	8.4
50	11.3	10.9	11.4
60	13.7	13.0	13.7

4. What is a standard curve? What can we determine using the standard curve we make in this lab?

Table 1.1: Chemicals and supplies

Chemicals	Supplies
Distilled water (DW)	10 mL pipette
NaCl (food salt without Iodine)	50 mL or 100 mL beaker (6)
	Spatula (1)
	Stirring rod (1)

Procedure:

Part A: Determine the density of water at room temperature

1. Rinse the given pipette and beaker with small amount of DW.

2. Use the scale and tare the empty clean and dry beaker and then use the pipet to add a 10.00 mL aliquot of DI water to the beaker, and record the mass of the water.

3. Measure the temperature of the water at room conditions and record it in table 2.3.

4. Repeat step-2 two more times for a total of 3 mass and volume measurements.

Part B: Make a standard curve

1. Place a clean, dry beaker on the scale and record the mass of the empty beaker in table 2.4.

2. Take the beaker in step 1 and label it as "standard solution 1", use a spatula to add solid NaCl to the beaker base on the below table and record the mass of the beaker +salt in table 2.4.

   Note: **The mass of NaCl does not need to be exactly the mass given in the table. But you do need to know exactly how much you measured out, so record the mass of the salt and beaker in table 2.4.**

   Table 1.2: Masses of NaCl to use.

Standard solution	NaCl mass (g)
1	2
2	1.5
3	1
4	0.67
5	0.33

3. Use the pipette and add 10 mL of DW to the beaker in step 2.

4. Mix the solution with the stirring rod until all of the solid has dissolved and record the mass of beaker + salt + water in table 2.4 and leave it a side.

5. Measure the temperature of your solution using the thermometer and record the reading in table 2.4.

6. Repeat step 3-5 for the rest of the standard solutions and record your data in table 2.4.

Note: record the temperature for each solution and clean your thermometer after each use.

Part C: Make a standard curve

1. Place a clean, dry beaker on the scale and record the mass of the empty beaker in table 2.4.

2. Measure 10 mL of your assigned unknown solution and transfer it to the beaker. Record your unknown number on your report sheet.

3. Measure the mass of both the 10.00 mL aliquot of the unknown and the beaker together and then record the value in table 2.4. Repeat two more times.

4. Record the temperature for your unknwon in table 2.4.

Report Sheet-1 of exp. 1: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the following tables

Table 1.3: Part A: Determine the density of water at room temperature

	Volume	Mass	Density	Temperature
Trial 1	10.00 mL			
Trial 2	10.00 mL			
Trial 3	10.00 mL			

Table 1.4: Part B: Make a standard calibration curve

Solution #	Volume (mL)	Beaker mass (g)	Beaker + salt mass (g)	Beaker + salt + water mass (g)	Calculated Salt mass (g)	Calculated Water mass (g)	Mass %= wt. salt/wt. water	Density	Temp. (°C)
1	10.00 mL								
2	10.00 mL								
3	10.00 mL								
4	10.00 mL								
5	10.00 mL								
unknown	10.00 mL		-------		--------	----------	From the graph		
unknown	10.00 mL		-------		--------	----------	From the graph		
unknown	10.00 mL		-------		--------	----------	From the graph		

Unknown number ( ): the concentration is ----------

Report Sheet-2 of exp. 1: (Attach any extra paper to this sheet)

Part B: Do the following calculations:

1. Calculate the average density of DI water at room temperature from your 3 sets of mass and volume data in Part I of the experiment. Show all of your work.

2. Use the internet to look up the value for density of water at the same temperature that you had. Make sure to cite your source. Compare the value of density to your measured value using a percent error calculation (assume the value your find from internet is more accurate value).
   % Error = $\frac{measured\ value - accurate\ value}{accurate\ value} \times 100$

3. Calculate the density and mass % of your 5 standard solutions.

4. Open Excel and create a column containing your mass % data and another column for density. Make a scatter chart plotting density data points as a function of mass % and then add a linear trendline. Make sure to display the trendline equation and R^2 value on your chart.

   Note: **submit a copy of your graph.**

5. Calculate the average density of your unknown NaCl solution from your 3 mass and volume measurements in Part III Step 3 of the experiment

6. Use the best fit line of your standard curve to calculate the mass % of NaCl in your unknown solution.

How confident are you in your unknown results?

Experiment 2: Calculating Molarity from % solutions

Objectives

1. Review the molarity unit expression for concentrations.
2. Review the use of the dilution equation.
3. Use the new concepts of units for expressing concentration.
4. help to determine what calculations to use and how to prepare solutions for your

Introduction

Solutions are a big part of biochemistry, biological and chemical based work. Stock solutions (or concentrated solutions) are often defined as percent, Molar concentration. If a solution is made of one component, then the molarity is usually shown.

Preparation of Solutions:

1. Molar solutions Molarity (M):

   Means the number of moles of solute per liter of solution. To prepare a 1 M solution. When all the solid is dissolved and the solution is at room temperature, dilute to the mark and invert the flask several times to mix.

2. Percent solutions:
   a. Mass percent means the number of grams of solute per 100 g of solution. For example, 10 g sodium chloride in 90 g water is a 10% by mass solution. mass percent = mass of solute/mass of solution = 10 g/(10 g + 90 g) × 100% = 10%
   b. Volume percent means the number of milliliters of solute per 100 mL of solution. The volume percent of a solution cannot be calculated directly from the volumes of its components because the final volume may not equal the sum of the components' volumes. To prepare volume percent solutions, first determine the final volume and concentration of solution desired and then determine the amount of solute. Dilute the solute in sufficient solvent to produce the final volume of solution desired. For example, to prepare 100 mL of a 10% by volume solution of acetic acid, dilute 10 mL acetic acid with distilled or deionized water to make 100 mL of solution.
   Note: Solutions of concentrated reagents, such as 37% hydrochloric and 85% phosphoric acids, are percent solutions by mass. In general, percent solutions are by mass.
3. Dilutions:
   When preparing a dilution, decide the volume and molar concentration of the resulting solution you require. Use the following equation to determine how much of the concentrated reagent is needed to prepare the diluted solution, $M_{reagent} \times V_{reagent} = M_{dilution} \times V_{dilution}$
   Where M is molarity and V is volume. Slowly add the calculated volume of concentrated reagent to the proper-size volumetric flask half filled with distilled or deionized water and swirl the flask to mix. Once the solution is at room temperature, dilute to the mark with water and invert the flask several times to mix. For example, what volume of 10 M acetic acid is required to prepare 1.0 L of 0.50 M acetic acid? 10 M × Vreagent = 0.50 M × 1.0 L Vreagent = 0.050 L = 50 mL 6 A volume of 50 mL of 10 M acetic acid is required to prepare 1.0 L of 0.50 M acetic acid.
5. Special cases:
   Often it is necessary to prepare solutions from chemicals that are less than 100% pure. To prepare solutions from these impure chemicals, first choose the volume and molarity of the resulting solution you require. Multiply the solution's volume by its molarity. The product (n) is the number of moles of pure chemical needed to produce that solution. $M_{pure} \times V_{pure} = n_{pure}$

Because the percent purity of chemicals sold commercially is measured by mass, first calculate the mass of the pure chemical needed to make the solution. Multiply the number of moles of pure chemical times the gram formula weight of the chemical.

$$\text{Mass of pure chemical} = n_{pure} \times \text{gram formula weight}$$

(1) mass of pure chemical = $M_{pure} \times V_{pure} \times$ gram formula weight

The mass of the impure chemical times the percent purity equals the mass of the pure chemical. Divide the mass of pure chemical by the percent purity to yield the mass of the impure chemical.

$$\text{Mass of impure chemical} \times \text{percent purity} = \text{mass of pure chemical}$$

(2) Mass of impure chemical = mass of pure chemical/percent purity Substitute the expression for mass of pure chemical from equation (1) into equation (2).

$$\text{Mass of impure chemical} = M_{pure} \times V_{pure} \times \text{gram formula weight/percent purity}$$

For example, what mass of potassium hydroxide that is 85.9% pure is needed to prepare 1.0 L of a 0.25 M solution of potassium hydroxide? The gram formula weight of potassium hydroxide is 56.11 g/mol. mass of impure chemical = Mpure × Vpure × gram formula weight/percent purity = 0.25 M × 1.0 L × 56.11 g/mol ÷ 0.859 = 16 g 7 If the chemical in question is a liquid, then one more calculation is required. Divide the mass of impure chemical by its density to yield the volume of chemical. volume of impure = mass of impure chemical in g/density of impure chemical in mL chemical in grams per milliliter Again, combine the previous equations. volume of impure chemical = Mpure × Vpure × gram formula weight/ (percent purity × density) For example, what volume of hydrochloric acid that is 37.1% pure is needed to prepare 1.0 L of a 0.10 M solution of hydrochloric acid? The gram formula weight of hydrochloric acid is 36.46 g/mol and the density of 37.1% hydrochloric acid is 1.200 g/mL. volume of impure chemical = Mpure × Vpure × gram formula weight/(percent purity × density) = 0.10 M × 1.0 L × 36.46 g/mol ÷ (0.371 × 1.200 g/mL) = 8.2 mL

6. Normal solutions:

Another concentration term sometimes used is normality. Normality (N) means the number of equivalents of solute per liter of solution. An equivalent is defined separately for acid-base and reduction-oxidation (redox) chemistry. In acid-base chemistry, an equivalent is the mass of chemical that donates or accepts one mole of protons. For example, sulfuric acid is a diprotic acid. One-half mole of sulfuric acid therefore provides one mole of protons. To prepare a 1 N solution of sulfuric acid, slowly add one-half gram formula weight of sulfuric acid to a clean 1-L volumetric flask and fill to the mark with distilled or deionized water. In redox chemistry, an equivalent is the mass of chemical that donates or accepts one mole of electrons. Determine the number of electrons a chemical donates or accepts from its half-reaction. For example, one mole of aluminum (III) reacts with three moles of electrons to give one mole of aluminum metal.

$$Al^{3+} + 3e– \rightarrow Al$$

To prepare a 1 N solution of aluminum (III), slowly add one-third gram formula weight of an aluminum (III) compound to a clean 1-L volumetric flask, and fill to the mark with distilled or deionized water

Pre-lab question

1. Describe how you would prepare 1 L of a 1 M solution of sodium chloride. The gram formula weight of sodium chloride is 58.44 g/mol.
2. Describe how you would prepare 100 g of a solution that is 0.5% phenolphthalein by mass.
3. Describe how you would prepare 1.0 L of a 0.10 M solution of sulfuric acid from a 3.0 M solution of sulfuric acid.

Table 2.1: Chemicals and supplies

Chemicals	Supplies
Distilled water	10 mL pipette (2)
Conc. HCl	25 mL volumetric flask (3)
NaCl	50 mL beaker (2)
10% NaOH	Stirring rod

Procedure:

Note: Students must show the instructor their calculation before starting the procedure.

Part A: Preparing 0.1 N HCl

1. Calculate the volume you need to take from the stock solution using the normality formula.

2. Use the pipette to measure the volume you calculate from step 1 and added to 25 mL volumetric flask and then

3. Use the distilled water to complete the volume to the mark.

Part B: Preparing 0.5 M NaCl

1. Calculate the weight of NaCl using the molarity formula.

2. Use 50 mL beaker to measure the amount of NaCl you calculate in step 1- part B using the scale.

3. Dissolve the weight in a reasonable amount of D.W then transfer it to another 25 ml volumetric flask and complete the volume with D.W to the mark.

Part C: Preparing 0.3 M from 10% (%W/W) NaOH

1. Calculate the concentration of NaOH in molarity unit from the 10% NaOH. (Density of 10% NaOH is 1.1111g/mL.

2. Use the dilution equation to find the amount of volume you need to take from the 10% NaOH solution.

3. Use a clean pipette to measure the volume you calculate in step 2- part C and transfer it a clean 25 mL volumetric flask and complete the volume with D.W to the mark.

Report Sheet of exp. 2: (Attach any extra paper to this sheet)

Group Names:

Part A: Collect your data by filling the following tables:

Table 2.2: Recording the calculated data

Part A: preparing 0.1N HCl					
Gram of HCl		Calculate Volume of HCl		Measured Volume of HCl	
Part B: preparing 0.5 M NaCl					
Grams of NaCl		Measured grams of NaCl		Volume used	
Part C: Preparing 0.3 M from 10% NaOH					
Conc. of NaOH (M) from the 10%		Calculated volume		Measured volume	

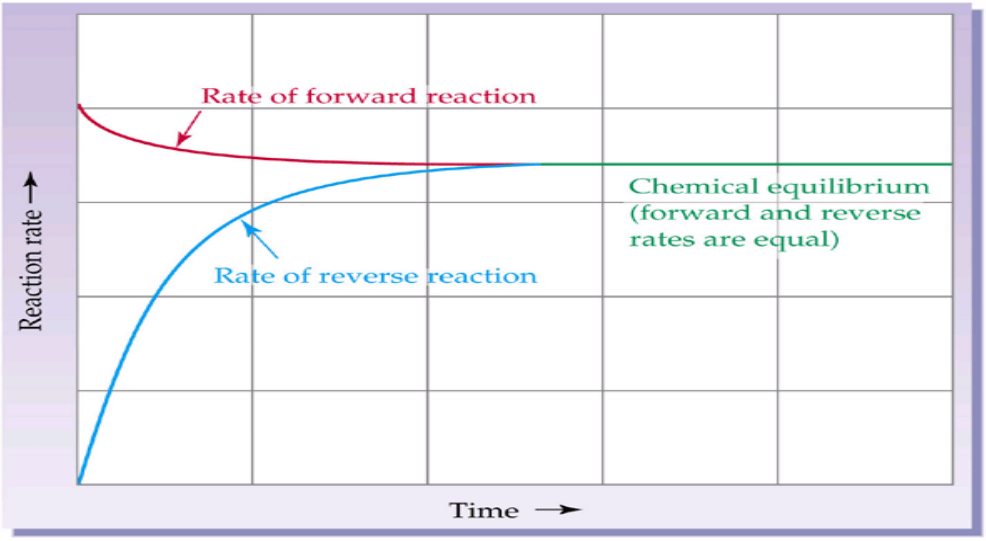
Part B: Answer the following:

1. Do the theoretical calculations in a separate sheet for part A, part B, and part C of your procedure and attach it with this sheet.

2. Describe how you would prepare 500 mL of a 0.25 M solution of sodium hydroxide from a 5.0 M solution of sodium hydroxide (read the introduction).

3. Describe how you would prepare 500 mL of a 1.0 M solution of potassium chloride that is 93.0% pure. The gram formula weight of potassium chloride is 74.56 g/mol. (read the introduction).

Experiment 3: Le Chatelier's Principle Effect of Concentration and Heat



Objective

Observe the effect of an applied stress like concentration and heat on a chemical system at equilibrium

Introduction

Reversible reaction is in which both the forward and the backward reactions occur simultaneously:

$A + B \rightleftharpoons C + D$

Le Chatelier's principle states that if an external stress is applied to a chemical system at equilibrium, the equilibrium will shift in the direction that will minimize the effect of the stress. In term of chemical systems there are three primary stressors: changes in concentration, changes in temperature and changes in pressure.

The Effect of Concentration

Consider a hypothetical reversible reaction already at equilibrium: $A + B \rightleftharpoons C + D$. If, for example, the concentration of A is increased, the system would no longer be at equilibrium. The rate of the forward reaction ($A + B \rightarrow C + D$) would briefly increase in order to reduce the amount of A present and would cause the system to undergo a net shift to the right. Eventually the forward reaction would slow down and the forward and the backward reaction rates become equal again as the system returns to a state of equilibrium. Using similar logic, the following changes in concentration are expected to cause the following shifts:

Increasing the concentration of A or B causes a shift to the right.

Increasing the concentration of C or D causes a shift to the left.

Decreasing the concentration of A or B causes a shift to the left.

Decreasing the concentration of C or D causes a shift to the right.

The Effect of Temperature

A change in temperature will also cause a reversible reaction at equilibrium to undertake a shift. The direction of the shift largely depends on whether the reaction is exothermic or endothermic. In exothermic reaction, heat energy is released and can thus be treated as a product. In endothermic reaction, heat energy is absorbed and thus be considered a reactant.

Exothermic $A + B \rightleftharpoons C + D + \text{heat}$

Endothermic $A + B + \text{heat} \rightleftharpoons C + D + \text{heat}$

Per Le Chatelier's principle, if the temperature is increased, a shift away from the side of the equation with "heat" occurs. If the temperature is decreased, a shift towards the side of the equation with "heat" occurs.

In this lab, the effect of applying stresses to variety of chemical systems at equilibrium will be explored. By observing the changes that occur (color changes, precipitate formation, etc.) the direction

of a particular shift may be determined. Such shifts may then be explained by carefully examining the effect of the applied stressors as dictated by Le Chatelier's principle.

Pre-lab questions

1. Define and describe chemical equilibrium
2. State Le Chatelier's Principle
3. List the three factors (stresses) that cause shifts in equilibrium

Table 3.1: Chemical and Supplies

Chemicals	Supplies
Solid NH_4Cl	Small test tube (10)
Saturated NaCl (prepare by TA)	Rack
Concentrated 12 M HCl	Test tube holder
0.1 M $CoCl_2$ (prepare by TA)	Bunsen burner
15M NH_3 (prepare by TA ammonia solution)	100 mL labeled Beaker beside each solution
Phenolphthalein indicator	10 mL labeled graduated cylinder beside each solution
0.1 M K_2CrO_4 (prepare by TA)	
6 M HNO_3 (prepare by TA)	
10% NaOH (prepare by TA)	

Safety Note

1. All of the acids and bases used in this experiment (NH_3, HCl, HNO_3, and NaOH) can cause chemical burns. In particular, concentrated 12 M HCl is extremely dangerous! If any of these chemicals spill on you, immediately notify your instructor.

2. Direct contact with silver nitrate ($AgNO_3$) will cause dark discoloration to appear on your skin. These spots will eventually fade after repeated rinses in water.

3. You will be heating a solution in a test tube directly in a fire source flame. If the solution is overheated it will splatter out of the tube, so be careful NOT to point the tube towards anyone while heating.

Note: Record all observations on your report. These should include, but not limited to color change and the formation of precipitates.

Procedure

Part I: Saturated sodium chloride (NaCl) solution

1. Place 3 mL of saturated $NaCl_{(aq)}$ into a test tube.
2. ***Carefully*** add concentrated 12 M HCl drop-wise to the solution in the test tube until a distinct change occurs. Record your observation.

Part II: Acidified chromate solution

1. Place 3 mL of 0.1 M K_2CrO_4 into another test tube.
2. Add 3 mL of 6 M HNO_3 to the solution in step 1. Record your observation.
3. Add the 10% NaOH drop-wise until the original color is returned (The chromate solution color). Record your observation.

Part III: Aqueous ammonia solution

1. Place 3 mL of the stock solution-1 into a new test tube.
2. Add a medium spatula of NH_4Cl powder to the solution in this test tube. Record your observation.

Part IV: Cobalt chloride solution

1. Place 3 mL of 0.1 M $CoCl_2$ into 3 new test tubes. Label these test tubes with numbers 1-3.
2. The solution in test tube #1 should remain untouched. It is a control to compare with test tube number 2 and 3.
3. To the solution in test tube #2, carefully add concentrated 12 M HCl drop-wise until a distinct color change occurs. Record your observation.
4. To the solution in test tube #3, first add a medium spatula full of solid NH_4Cl. Firmly hold test tube #3 with the holder and waft it back and forth through the flame (to prevent overheating and bumping) for about 30 seconds, or, until a distinct change occurs. Record your observation. Then cool the solution in test tube #3 back to room temperature by holding it under running tap water, and again record your observation.

Report Sheet of Exp. 3: (Attach any extra paper to this sheet)

Group Names:

Part A: Collecting Data

Table 3.2: Recording the reaction observations.

Part I: Saturated sodium chloride solution	
Adding 12 M HCl	
Part II: Acidified chromate solution	
Adding 6 M HNO_3	
Add the 10% NaOH	
Part III: Aqueous ammonia solution	
Solid NH_4Cl	
Part IV: Cobalt chloride solution	
Test tube #2, adding 12 M HCl	
Test tube #3, adding solid NH_4Cl	
Heating	
Cooling	

Part B: Write a balance chemical equation for each part.

Part C: Answer the following questions

1. Explain how Le Chatelier's principle applied for part 2.

2. What will happen if you add HCl to the Ammonia solution in part 3?

1. Explain any weak or strength point you noticed while you are conducting the experiment?

2. In your Opinion, how we can update (improve) this experiment?

Experiment 4: Hydrogen Phosphate Buffer Systems

Objectives:

1. You will prepare a buffer solution with an assigned concentration and pH.
2. Study the effect of dilution on the buffer system.

Introduction

Buffers are fundamental to wet chemistry, although the basic idea of buffers extends far beyond solutions. The primary idea behind a buffer is to dampen or minimize the effects of changes to or within the system so that the impact on the system is not as bad. There are buffers in electrical systems, irrigation systems, computers, and mechanics. Shock absorbers, for instance, prevent you from feeling bumps in the road when you are driving. Similarly, in wet chemistry, buffers can help reduce or minimize external stresses (changes in temperature or pressure), or chemical reactions from changing the overall pH of a solution. It is critical to select a buffer that is well suited to the system you are studying. Will the temperature of the system be changing? Will the pressure be changing? How does a change in either affect the pKa of the buffer? What is the pH value you would like to maintain? A buffer is most effective when the pH of the solution is in the vicinity of its pKa value (±1 pH unit). In this laboratory, you will learn how to calibrate and use a pH meter and *you will be preparing buffer systems Sorensen's Buffer*.

An acid will dissociate in water to a conjugate base and proton. Consequently, acids are typically thought of as proton donors:

$$\underset{\text{Acid}}{[HA]} \overset{K_a}{\leftrightarrow} \underset{\text{Proton}}{[H^+]} + \underset{\text{Conjugate Base}}{[A^-]} \qquad \text{---------- (1)}$$

Ka is the equilibrium constant that determines the extent that the acid will dissociate in water:

$$K_a = \frac{[H^+][A^-]}{[HA]} \qquad \text{---------- (2)}$$

Recall that the pH of a solution in water is the negative log of the concentration of hydrogen ions, and is a more convenient way to express tiny concentrations. Similarly, the pKa is also the negative log of the equilibrium constant Ka:

$$pH = -\log[H^+], \text{ or alternatively, } 10^{-pH} = [H^+] \qquad \text{---------- (3)}$$

$$pK_a = -\log(K_a), \text{ or alternatively, } 10^{-pKa} = K_a \qquad \text{---------- (4)}$$

By substituting Equations (3) and (4) into Equation (2), we can derive the Henderson-Hasselbalch equation:

$$10^{-pKa} = \frac{10^{-pH}[A^-]}{[HA]} \qquad \text{---------- (5)}$$

$$\frac{[HA]}{[A^-]} = \frac{10^{pKa}}{10^{pH}} \qquad \text{---------- (6)}$$

$$\frac{[HA]}{[A^-]} = 10^{pKa-pH} \qquad \text{---------- (7)}$$

According to the Henderson-Hasselbalch buffer relationship, pH, pKa, and the buffer component concentrations for a weak acid are related as follows:

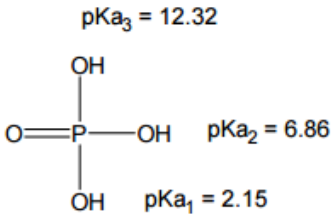
$$\frac{[acid]}{[base]} = 10^{pK_a-pH} \qquad \text{---------- (8)}$$

Here, the 'acid' is the proton donor (HA), and the 'base' is the conjugate base (A-) in Equation (1). This is a very convenient form of the equation, because it allows us to see the following:

- If the pKa is greater than the pH, there will be more of the acid form in the solution.
- If the pKa is equal to the pH, there will be an equal amount of acid and base in the solution.
- If the pKa is less than the pH, there will be more of the base form in the solution.

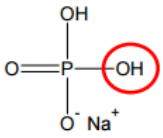
Pairing a Weak Acid with its Salt: Sorensen's Buffer

Many buffer systems are weak acids paired with their respective salts. One example is citric acid paired with sodium citrate. The reason that two solutions are made at the beginning – a solution of the acid of the buffering agent, and another solution of its salt – is so that we may titrate one with the other to attain the exact pH we are looking for. It is assumed that when the salt of a buffer is dissolved in water, it will dissociate completely and go into solution in the ionic form. It is important to note however that a buffering system can be as simple as a weak acid added to de-ionized water, with the pH of solution adjusted close to the pKa using either NaOH or HCl. Buffering systems are not limited to weak acids, they can also be weak bases (e.g. ammonia + ammonium chloride). Weak bases may be used for solutions where the pH desired is above 7. We will focus our discussion on weak acid buffers paired with salts of their conjugate bases. Since it was already stated a buffer is most effective within 1 pH unit of its pKa, you would think that a given buffer would only be useful in the vicinity of one pH value. However, many buffers have more than one acidic group attached, which vary in affinity to their respective protons. Sorensen's Buffer (phosphate buffer) has three acidic groups, each with different pKa values (see right panel).

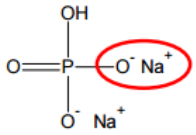


Phosphoric Acid

This makes Sorensen's buffer useful in the pH ranges 1.15 – 3.15, 5.86 – 7.86, and 11.32 – 13.32. The second pKa is close to 7, and so Sorensen's buffer is typically used for buffer systems at pH 7. Since we would like to make use of the second pKa of phosphate, we might as well choose the weak acid and corresponding salt of the conjugate base of the second acidic group:



Sodium Phosphate Monobasic
weak acid



Sodium Phosphate Dibasic
salt of conjugate base

The monobasic acid dissociates into its conjugate base, and thus becomes dibasic. (It's called "basic" since the charged –O- form is the acid's conjugate base. Confused yet?). It will do this depending on the pH of the solution, according to the Henderson-Hasselbalch equation.

A buffer is usually prepared in concentrations ranging from 0.1 – 10 M. The way that a buffer works, is that provided there are both forms (acid and conjugate base) of the buffer present (i.e. the pH is around the pKa), then if another acid dissociates to add a proton to solution, the proton will be absorbed by the buffer's conjugate base instead of lowering the pH. If a base is added to the solution, it will result in a hydroxide ion, which will in turn react with the buffer's weak acid instead of raising the pH. In this way, the balance of hydrogen ions is protected, and changes in pH are much smaller than they would have been in the absence of buffer.

Pre-lab question

1. Acetic acid has a K_a value of 1.8×10^{-5}. How many grams of $NaC_2H_3O_2$ would have to be added to 100.0 mL of 0.10 M $HC_2H_3O_2$ to prepare a buffer with a pH of 4.50?
2. The pH of a solution is measured to be 3.50. Calculate the hydrogen ion concentration?

Table 4.1 Chemicals and Supplies

Chemicals	Supplies
0.1 M Dipotassium hydrogen phosphate, K_2HPO_4	pH-meter
0.1 M Potassium dihydrogen phosphate, KH_2PO_4	50 mL graduated cylinder
pH= 7.0	250 mL volumetric flask (2)
pH= 4.0	100 mL beaker (2)
	250 mL beaker (1)
	Disposable pipette (dropper)
	Parafilm

Procedure

Part I: Calibrating the pH-meter **(Lab assistant should prepare this part only)**

1. Place 50 mL of distilled water in a clean dry 100 mL beaker and measure its pH.

2. Place the electrode into Standard Solution at pH 7.00. Turn on the meter and wait for 1-2 minutes while swirling the electrode gently in the solution. Record pH reading.

3. Calibrate the pH meter as closely as possible for pH = 7.00 by adding an acid or a base.

4. Repeat Step 1-3 for Standard Solution at pH 4.00.

Note: Be sure to rinse the tip of the pH-meter with distilled water

Part II: Preparing Sorensen's buffer solution for the assigned pH:

For making 0.1 M Sorensen's buffer at pH 6.81:

1. Use a clean beaker and measure 4.35 g of K_2HPO_4.

2. Add a reasonable amount of water to the beaker in step 1, when all the solid dissolve, transfer the liquid to 250mL volumetric flask and complete the volume with distilled water to the mark.

   Note: Label the volumetric flask as (A^-).

3. Repeat steps 1-2 but by measuring 3.4 g of KH_2PO_4.

   Note: Label the volumetric flask in step 3 as (HA).

4. To prepare a buffer solution from acid-base conjugate pairs:

   a. From flask **(A^-)**, measure 178.7 mL using a graduated cylinder and transfer this measurement to a clean 250 mL beaker.

      Note: Label this beaker as (buffer).

   b. Bring a burette and rinse it with about 10 mL of solution **(HA)**. Then fill your burette with this solution to the mark.

   c. Put the **buffer** beaker on a hot-plate and place a piece of magnet bar in it and set mixing at low speed.

      Note: do not allow the stir bar to hit the pH meter, or a vortex to appear

   d. Insert the pH electrode inside the **buffer** beaker and make sure the tip of the pH electrode is submerged and does not touch the bottom of the beaker.

   e. Start adding **(HA)** solution that exist inside the burette to the solution **(A^-)** in your beaker. You will stop the addition until the pH meter read the desired pH which is 6.81.

   f. Record the volume of (HA) solution that been used in table 4.2.

5. Seal your buffer solution with parafilm, label it with the group name, and store it for experiment 5.

Part III: Determining the effect of diluting the buffer on the pH

1. Repeat part II step 4-(a-f)

2. Remove the pH meter, look at the volume in your beaker, read it and add to the beaker the exact amount you read of distilled water.

3. Place the pH meter again and read the pH value.

4. Clean your mess.

Report Sheet of Exp. 4: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the table below

Table 4.2: Collecting data

Part II: Preparing Sorensen's buffer solution for the assigned pH			
	Measured pH	Theoretical pH	
Buffer solution		6.81	
Volume of HA used		Calculated volume of HA	
Part III: Determining the effect on the pH after diluting the buffer			
	Measured pH	Theoretical pH	
dilute buffer solution		6.81	

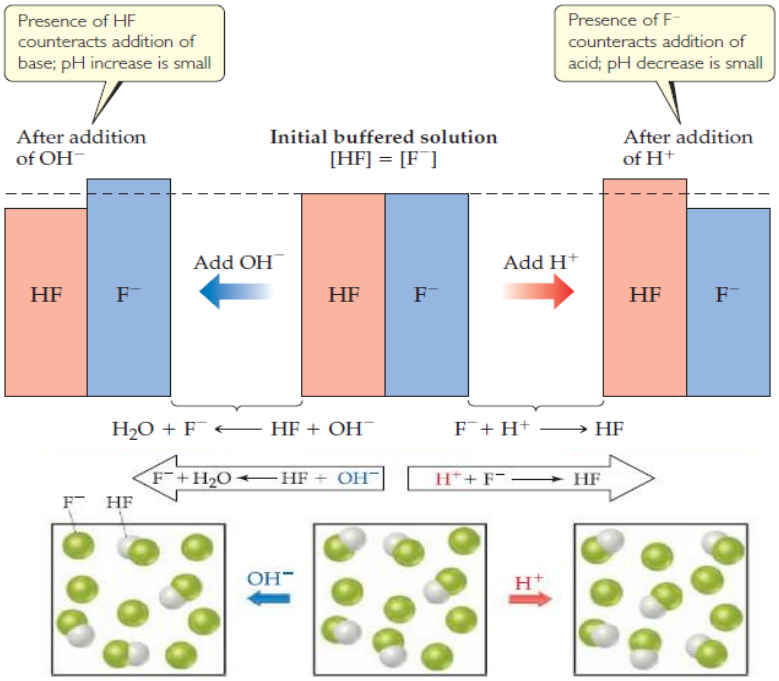
Part B: Show the protocol for preparing 250 mL of 0.1 M phosphate buffer, at pH 6.81

Part C: Answer the following

1. Compare between the theoretical pH value (calculated) and the experimental pH values measured in lab using the pH-meter.

2. If the buffer is dilute, will that effect on the pH value of the buffer? Explain.

Experiment 5: Measuring the Buffer Capacity



Objective

Determine the buffer capacity of a phosphate buffer solution.

Introduction

A buffer solution is one that is resistant to change in pH when small amounts of strong acid or base are added. For example, when 0.01 mole of strong acid or base are added to distilled water, the pH drops to 2 with the acid and rises to 12 with the base. If the same amount of acid or base is added to an acetic acid – sodium acetate buffer, the pH may only change a fraction of a unit.

Buffers are important in many areas of chemistry. When the pH must be controlled during the course of a reaction, the solutions are often buffered. This is often the case in biochemistry when enzymes or proteins are being studied. Our blood is buffered to a pH of 7.4. Variations of a few tenths of a pH unit can cause illness or death. Acidosis is the condition when pH drops too low. Alkalosis results when the pH is higher than normal.

Two species are required in a buffer solution. One is capable of reacting with – OH and the other will react with H 3 O + . The two species must not react with each other. Many buffers are prepared by combining a weak acid and its conjugate (acetic acid and sodium acetate) or a weak base and its conjugate (ammonia and ammonium chloride). In general, the pH range in which a buffer solution is effective is +/- one pH unit on either side of the pKa. The Henderson–Hasselbalch provides the information needed to prepare a buffer.

$$Ph = PkA + \log\frac{[conjugatebase]}{[weakacid]}$$

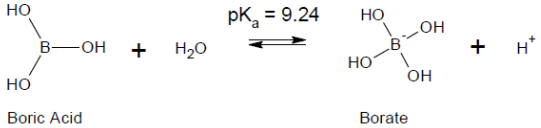
There is a limit to the amount of acid or base that can be added to a buffer solution before one of the components is used up. This limit is called the buffer capacity and is defined as the moles of acid or base necessary to change the pH of one liter of solution by one unit.

$$\text{Buffer Capacity} = \frac{(\text{number of moles of } ^-OH \text{ or } H_3O^+ \text{ added})}{(\text{pH change})(\text{volume of buffer in L})}$$

In this experiment, the buffer capacity of the buffer solution will be determined

Pre-lab question

1. Calculate the buffering capacity ratio $\frac{[base]}{[acid]}$ of buffer solution you prepared in experiment 4.
2. Borate buffer is used in gel electrophoresis. It is a combination of Boric Acid (MW 61.83g/mol), titrated with NaOH to the desired pH.



   a. What is the useful pH range of borate buffer?
   b. Create a protocol for preparing 500 mL of 0.1 M Borate buffer, at pH 8, assuming you already have a solution of 0.2 M NaOH. How will you prepare your stock solutions? How much of each will you require?

Table 5.1 Chemicals and Supplies

Chemicals	Supplies
Buffer solution from experiment 4	pH-meter
0.1 M NaOH	50 mL volumetric flask (1)
	10 mL pipette (1)
	50 mL beakers (1)
	Burette (1)
	Hot-plate and magnet bar

Procedure

Part I: Find the buffer capacity of the prepared buffer solution

1. Tare a clean beaker and measure 0.20 g NaOH.

2. Add a reasonable amount of water until all the solid dissolve then transfer the solution to 100 mL volumetric flask and complete the volume to the mark.

3. Set up a burette (Figure 1). Rinse and fill the burette with 0.10 M NaOH solution.

4. Use a clean pipette, measure 10.0 mL of buffer solution that you prepared in experiment 4 and transfer it to a clean 50 mL beaker.

5. Add 10.0 mL distilled water to the beaker in step 4 and put the **buffer** beaker on a hot-plate and place a piece of magnet bar in it and set mixing at low speed.

   Note: do not allow the stir bar to hit the pH meter, or a vortex to appear

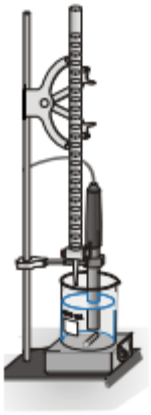


Figure 1

6. Insert the pH electrode inside the **buffer** beaker and make sure the tip of the pH electrode is submerged and does not touch the bottom of the beaker. (figure 1)

7. Measure the pH of your buffer again.

8. Begin adding the NaOH to the buffer solution in small increments, i.e. 0.05-0.1 mL. Record the total volume of NaOH added that cause the pH to risen one unit and record the pH of the solution in table 5.2.

9. Repeat step 4-8 two more times as the lab time allows.

Report Sheet of Exp. 5: (Attach any extra paper to this sheet)

Group Names:

Part A: Collecting data

Buffer type Prepared: _____

Concentration: _____

pH of buffer solution _____

Table 5.2: Fill the table below

	Trial 1	Trial 2	Trial 3
Volume of Buffer (mL)			
Volume of Buffer (L)			
Volume of 0.10 M NaOH (mL)			
Mole of NaOH			
Initial pH of Buffer			
pH after adding NaOH			
Change in pH			
Buffer capacity (β)			

Part B: answer the following:

1. Show your calculations to one of the three trials.

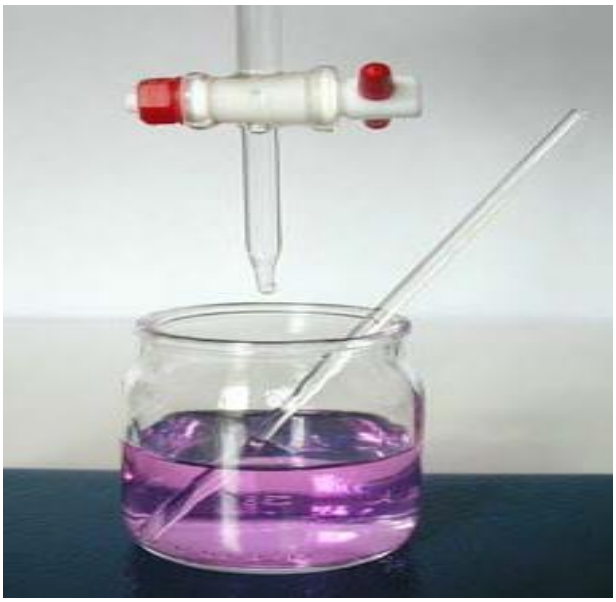
2. Calculate the buffer capacity using the following formula:
   Buffer Capacity, (β) = $\frac{(number\ of\ moles\ of\ -OH\ or\ H+\ added)}{(change\ in\ pH)(volume\ of\ buffer\ in\ L)}$

3. Find the average buffer capacity.

4. How does the concentration of the buffer affect the buffer capacity?

5. What differences would be observed if HCl were used in place of NaOH?

Experiment 6: Determining the Concentration of Citric Acid in 7-Up Using Titration



Objectives

Students will be able to

1. Learn the titration technique.
2. Determine an unknown concentration from a known concentration
3. Recognize the type of acids: monoprotic acids, diprotic acids, polyproticacids, weak acids, and strong acids.

Introduction

Titration is the method of determining the concentration of a solution by adding a carefully measured volume of a base of a known concentration to a carefully measured volume of acid of an unknown concentration, or vice versa, and using the knowledge of stoichiometry and acid-base neutralization reaction, we can determine the concentration of the unknown.

Acids can be classified as monoprotic, diprotic, and triprotic acids. A monoprotic acid has one proton that can undergo a reaction with a base, a diprotic acid has two protons, and a triprotic acid has three. Any acid that has more than one proton is called a polyprotic acid.

Citric acid is a weak, polyprotic acid that undergoes the following reaction:

$H_3C_6H_5O_{7(aq)} + NaOH_{(aq)} \longrightarrow Na_3C_6H_5O_{7\,(aq)} + 3HOH_{(l)}$

In this experiment you will be performing a titration to determine the concentration of citric acid in 7-Up. Prior to the titration the majority of the carbonic acid is removed by allowing the 7-Up to go flat so we do not have to take it into consideration.

The base is placed in the burette, so that a precise amount of solution can be added to the acid. The burette's precision is attributed to its graduation up and down the tube, making it one of the more expensive pieces of glassware in the lab.

The burette is used in a titration to dispense measured increment of one solution into a known volume of another solution. Only careful technique will allow you to detect the point where the reaction is complete; in this case, when all of the citric acid has been reacted with the base. This point is called the *equivalence point*. The *end point* is the point where the indicator being used changes color (also '*indication point*').

The technique of titration can be applied to other types of reactions such as oxidation-reduction, precipitation, complexation and other acid-base neutralization reactions.

Pre-lab questions

1. You found you needed an average volume of 25 mL of 0.05 M NaOH to neutralize the 7-Up. Calculate the molarity of the citric acid in the 7-Up. Show your work! (Read procedure for acid volume.)

2. What is the molar concentration of (H_3O^+) in a solution whose pH is equal to 2?

3. List two types of food that has an acidic or basic taste:

   Acids: _____

   Bases: _____

4. What is the difference between equivalence-point and end-point?

Table 6.1: Chemicals and supplies

Chemicals	Supplies
0.5 M KHP (prepare by TA)	50 mL burette
0.5 M NaOH (prepare by TA)	500 mL Erlenmeyer Flask
Phenolphthalein Indicator	50 mL graduated cylinder (2)
7-Up (decarbonized)	250 mL Beaker (2)
	Funnel

Safety Note

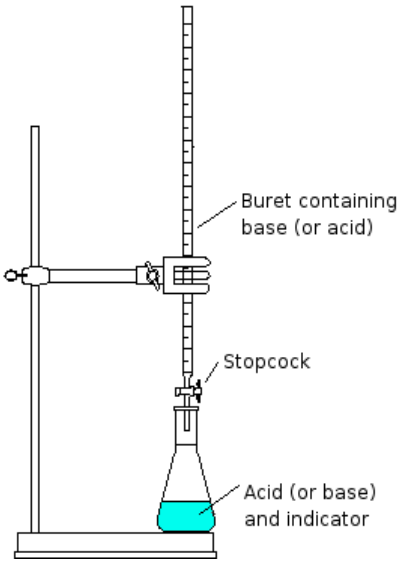
1. The acid and base used in this experiment (KHP and NaOH) can cause chemical burns. If any of these chemicals spill on you, immediately wash your hands for 15 minutes with soap. Also keep your hands away from your face.
2. Dispose the chemicals into the proper container.

Procedure

Part A: Preparing the equipment.

1. Rinse your burette with distilled water.
2. Use the burette clamp to place your burette on the stand.
3. Rinse your burette twice with 10 mL KHP solution that you will be standardizing by rotating the burette slowly, so that all the surfaces contact the solution.

   Note: make sure the stopcock is closed.
4. Drain this solution is step 3 into 250 mL beaker as a waste beaker by opening the stopcock**.**



5. Close the stopcock again and place the funnel in the top of the burette then carefully fill the burette to the mark with the KHP solution.
6. Open and close the stopcock to force the air bubbles out of the tip.
7. Record the initial volume of KHP in table 6.2 to the nearest hundredth milliliters.

Part B: Standardization of NaOH

1. Using the graduated cylinder, measure 25 mL of NaOH solution and transfer it to the Erlenmeyer flask.
2. Add 2-3 drops of phenolphthalein indicator, (Ph-Ph), to the Erlenmeyer flask.

   Note: make sure that you record the initial volume of the KHP in part A.
3. Slowly add the KHP solution to the NaOH solution until the pink color disappears.
4. Read your final volume from your burette. Record your data in table 6.2

Part C: Titration of 7-Up

1. Full the burette to the mark with standard NaOH.
2. Measure 10 mL of 7-Up and pour it into a clean Erlenmeyer flask.
3. Add 2 drops of phenolphthalein indicator, (Ph-Ph), to the Erlenmeyer flask.
4. Start the titration by adding slowly NaOH to your 7-Up. You will stop when the color change to a very faint pink.
5. Repeat this part two more times.

Report Sheet of Exp. 5: (Attach any extra paper to this sheet)

Group Names:

Part A: Collecting Data

Table 6.2: Recording data collected from the procedure.

Standardization of NaOH	
Volume of NaOH	25 mL
Initial burette reading of KHP, V*i*	
Final burette reading of KHP, V*f*	
Total volume, V*f* – V*i*	
Molarity of NaOH	

Titration of citric acid in 7-Up			
	Trial 1	**Trial 2**	**Trial 3**
Volume of 7-Up	10 mL	10 mL	10 mL
Initial burette reading of NaOH, V*i*			
Final burette reading of NaOH, V*f*			
Total volume ΔV = V*f* – V*i*			
Average ΔV			
Molar mass of citric acid			

Part B: Do the following Calculations

Calculate:

1. The molarity of standard NaOH solution using the formula $M_a * V_a = M_b * V_b$

2. The Average molarity of the citric acid in 7-Up

Part C: **Answer the following:**

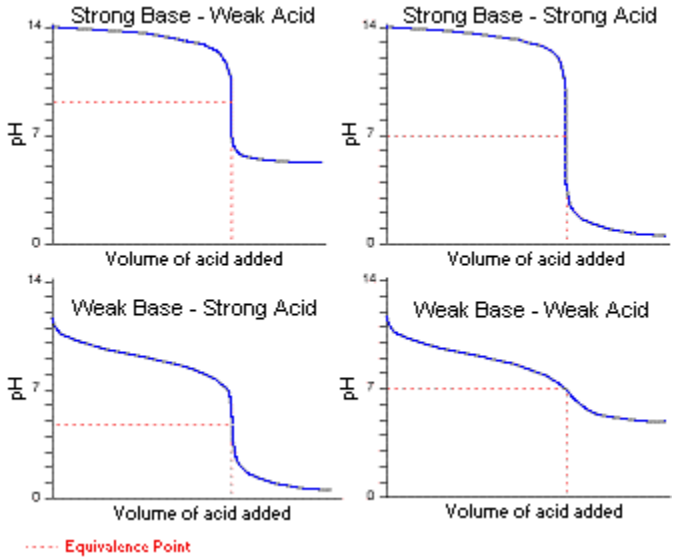
1. Based on the concentration of Citric acid you calculate for 10 mL, is it safe to drink a 7-Up can that contain 250 mL citric acid?

2. Which type of acid is the citric acid?

3. What will happen if we did not use the indicator?

4. What is the difference between equivalent point and End-point?

Experiment 7: Titration Curve of Strong Acid with Strong Base



Objectives

1. Students will conduct two titrations, one of a Strong acid with a strong base and the other is a weak acid with a strong base
2. Determine the Ka value of the weak acid using the constructed titration curve.

Introduction

An acid/base titration can be monitored with an indicator or with a pH meter. In either case, the goal is to determine the equivalence point of the titration.

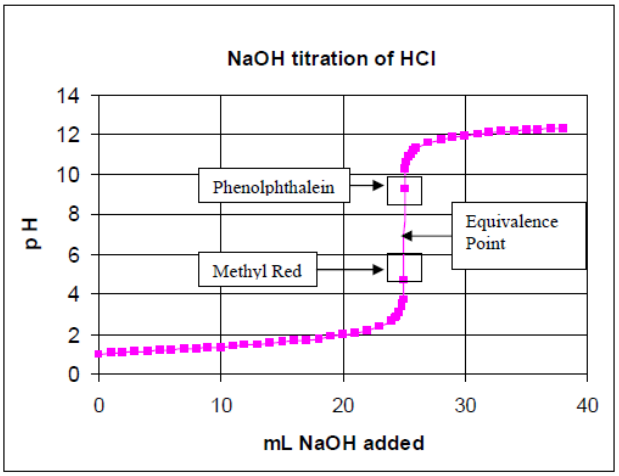


Figure 1. Titration of 25.0 mL of 0.1M HCl by 0.1 M NaOH. Blocked areas on the curve indicate the pH range in which phenolphthalein and methyl red change colors.

This is the point at which enough titrant has been added to the analyte to just exactly neutralize the analyte. In this experiment, knowledge of the equivalence point will be used to obtain information about the acid dissociation constant, $K_{a,}$ of the acid being titrated.

When an indicator is used in a titration, the color change occurs at what is called the endpoint. If the indicator has been properly selected, this point will be the same as the equivalence point. When a pH meter is used, the pH of the solution is recorded as the titrant is added. The pH versus the volume of titrant added can be plotted on what is called a *titration curve.* In this case the equivalence point occurs at the point where very small additions of titrant cause a very rapid rise in the pH. Graphically, it is also the point on the curve where the slope, $\Delta pH/\Delta V$, changes from positive to negative (called the *inflection point*.)

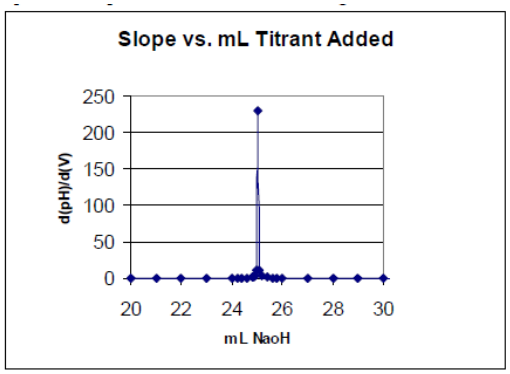


Figure 2. Plot of slope, $\Delta pH/\Delta V$, vs. mL NaOH from 20 to 30 mL based on Figure 1 titration.

Figure 1 is a titration curve for the titration of HCl by NaOH, a strong acid and strong base, where 25.0 mL of 0.1 *M* HCl is titrated with 0.1 *M* NaOH.

Note that the slope, $\Delta pH/\Delta V$, becomes large when the volume of NaOH added is at 25 mL, so this is the equivalence point. Because of this rapid rise through a range of pH values when the equivalence point is reached, a wide variety of indicators may be used to detect the endpoint visually. Either methyl red or phenolphthalein can be used for an HCl/NaOH analysis, since both will exhibit color changes in the range of pH values at the equivalence point. Also note that if the slope, $\Delta pH/\Delta V$, is plotted versus the volume of titrant added, the inflection point will appear as a spike. This is the most precise method for determining the equivalence point, and it is shown in Figure 2 below.

Pre-Lab questions:

1. Consider 25.0 mL of a 0.10 M strong acid solution and 25.0 mL of a 0.10 M weak acid solution, both titrated with a 0.20M KOH(aq) solution. List at least three differences between the appearances of the titration curves for the two different acid solutions.
2. What happens to the Ka value when the pH decrease?
3. What is the volume you added in each part to reach the equivalence point?
4. What is the difference between the equivalence point and the end point?

5. Can you suggest another indicator to be used in this experiment? Why you select this indicator?

Table 7.1: Chemicals and Supplies

Chemicals	Supplies
Distilled water	pH meter
0.1 M HCl (prepare by TA)	100 mL beaker (2)
0.1 M NaOH (prepare by TA)	25.0 mL Pipet
	50.0 mL Burette
	50 mL graduated cylinder
	Hot plate (for stirring)

Safety Note

1. The acid and base used in this experiment (KHP and NaOH) can cause chemical burns. If any of these chemicals spill on you, immediately wash your hands for 15 minutes with soap. Also keep your hands away from your face.

2. Dispose the chemicals into the proper container.

Procedure

Part I: Strong acid-Strong Base titration.

1. Obtain a clean, dry 100-mL beaker and label it as rxn.

2. Using the pipette, withdraw 25.0 mL of 0.1 M HCl solution and transfer it to the 100-mL rxn beaker in step 1.

3. Using the pH meter, record the pH of the solution in step 2.

4. Add 1-2 drops of (ph-ph) indicator to rxn beaker and leave it a side.

5. In another 100-mL beaker (label it B), Obtain 75.0 mL of 0.1 M NaOH solution.

6. Rinse the burette with the standard solution two times by adding 2 mL of NaOH in each time. Swirl the NaOH around the burette and discard into the sink.

   Note: Make sure the stopcock is closed when you rinse your burette with the base but open it when you discard the waste.

7. Using the funnel, carefully add the 0.1 M NaOH to the burette (Make sure the stopcock is closed). Go about an inch past the top line on the burette and be careful not to let it overflow.

8. With beaker B under the burette, slowly bring the miscues to the zero mL line or below.

9. Add 5.0 mL of NaOH to the rxn beaker, stir the solution and record the new pH after this addition.

Note: put a white sheet paper under the rxn beaker that you can see the change in the color clearly.

10. Continue the titration in step 9 for by reaching to the following additions and measure the pH for each 5.0 mL increment: 10.0 mL, 15.0 mL, 20.0 mL, 25.0 mL, 30.0 mL, 35.0 mL of NaOH.

11. Wash out the rxn beaker and refill the burette with NaOH for the next titration

Note: For calculating your data you need to read part II of this experiment.

Part II: Using Gran's plot graph to collect data

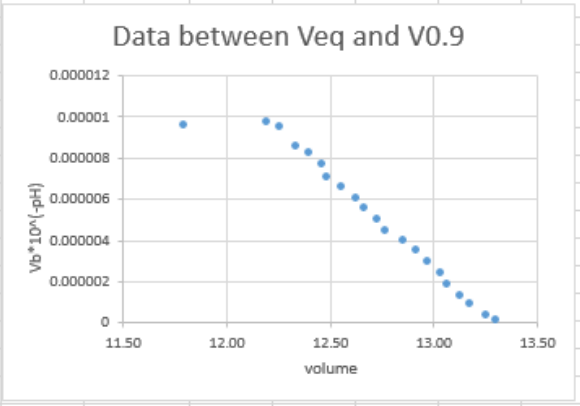
1. Assume you collect some data from titration as given to you below, use the last 10-20% volume of the equivalence point, this mean we are going to use 80-90% volume of the equivalence. For example if the volume at equivalence point was 13.3 then you need to take 90% of that volume:
   13.30 mL 0.9 = 11.97 mL

2. We can see that the volume (11.97 mL) we got is not among the data but we have a value close to it which is (11.80 mL) which we can call it (V0.9).

3. We select and copy the data between (V0.9) and (Veq) and then past the in a separate columns.

4. In a new column we use the equation: $Vb*10^{\wedge(-pH)}$ and graph the volume of the base versus the column of this equation values.

volume	pH	b*10^(-pH)	
11.80	6.09	9.6E-06	we can delet this value
12.20	6.10	9.7E-06	
12.26	6.11	9.5E-06	
12.34	6.16	8.5E-06	
12.40	6.18	8.2E-06	
12.46	6.21	7.7E-06	
12.49	6.25	7E-06	
12.56	6.28	6.6E-06	
12.63	6.32	6E-06	
12.67	6.36	5.5E-06	
12.73	6.41	5E-06	
12.77	6.46	4.4E-06	
12.86	6.51	4E-06	
12.92	6.57	3.5E-06	
12.97	6.65	2.9E-06	
13.04	6.74	2.4E-06	
13.07	6.85	1.8E-06	
13.13	7.00	1.3E-06	
13.18	7.16	9.1E-07	
13.26	7.56	3.7E-07	
13.30	8.28	7E-08	



	volume	pH
	0.00	4.06
	1.50	4.39
	3.00	4.64
	4.50	4.86
	6.00	5.04
	7.50	5.23
	9.00	5.44
	9.40	5.50
	9.80	5.57
	10.20	5.65
	10.62	5.74
	11.00	5.82
	11.40	5.95
V0.9	11.80	6.09
	12.20	6.10
	12.26	6.11
	12.34	6.16
	12.40	6.18
	12.46	6.21
	12.49	6.25
	12.56	6.28
	12.63	6.32
	12.67	6.36
	12.73	6.41
	12.77	6.46
	12.86	6.51
	12.92	6.57
	12.97	6.65
	13.04	6.74
	13.07	6.85
	13.13	7.00
	13.18	7.16
	13.26	7.56
Veq	13.30	8.28
	13.36	8.85
	13.43	9.16
	13.48	9.18
	14.48	10.66

5. We can delete unwanted value that is outlier from the linear behavior.

Report Sheet of Exp. 7: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the table below

Table 8.2: collecting data:

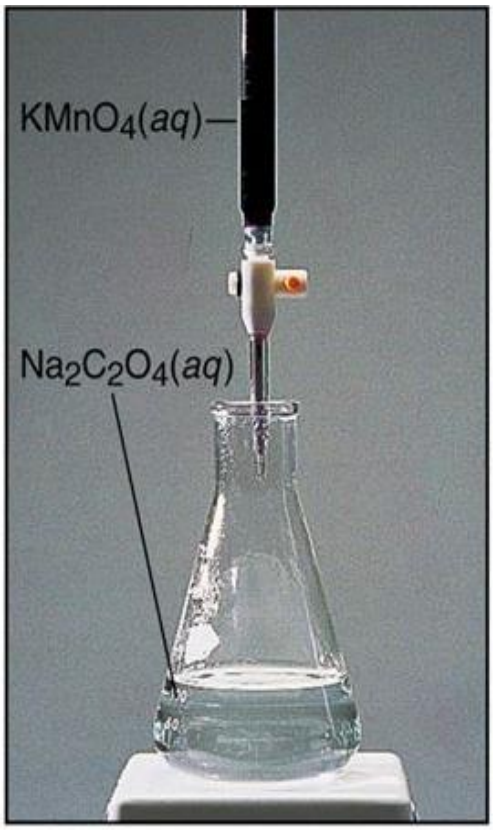
Volume of 0.1 M NaOH (mL)	Measured pH	Theoretical calculated pH
0.00		
5.0		
10.0		
13.0		
15.0		
17.0		
19.0		
20.0		
21.0		
22.0		
23.0		
24.0		
24.2		
24.4		
24.6		
24.8		
25.0		
27.0		
29.0		
30.0		
32.0		
34.0		
36.0		

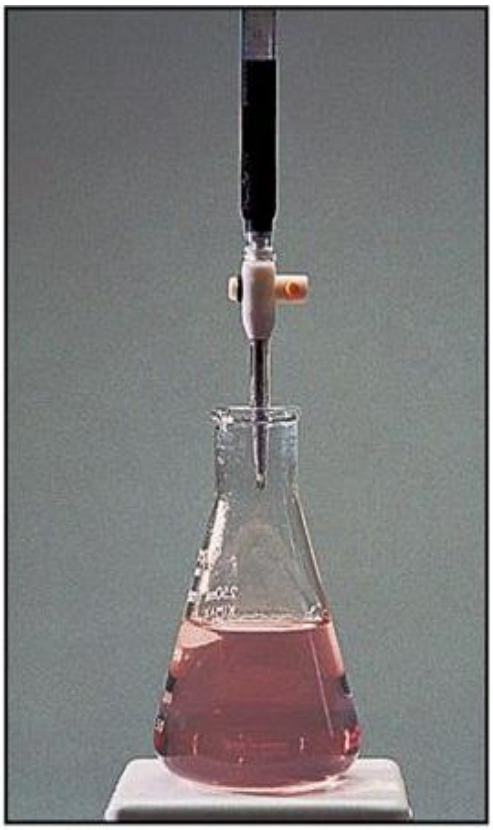
Part B: Calculation the following:

1. Calculate the theoretical pH value for each addition and compare it with the practical pH.

2. Use Excel sheet to graph the pH verses the NaOH volumes. Use part II of the procedure and the introduction for using the Excel sheet in experiment 2 for this purpose and find the equation of the straight line and solve for X which represent the accurate equivalence point

Experiment 8: Redox Titration





Objective

1. Practice the normality term in preparing a solution of an oxidizing agent,
2. Standardize the oxidizing agent solution by titrating it with standard solution of a reducing agent.
3. Use the standard solution to find an unknown concentration.

Introduction

Oxidation-reduction ("redox") titration is a type of titration based on the loss or gain of electrons between the analyte and the titrant. The endpoint of this titration is where the # of equivalence of the reducing agent is equal to the number of equivalence of the oxidizing agent. Potassium permanganate, $KMnO_4$, is a strong oxidizing agent. Permanganate, MnO_4^- , is an intense dark purple color. Reduction of purple permanganate ion to the colorless Mn^{+2} ion, the solution will turn from dark purple to a faint pink color at the equivalence point. No additional indicator is needed for this titration. The reduction of permanganate requires strong acidic conditions. In this experiment, permanganate will be reduced by oxalate, $C_2O_4^{2-}$ in acidic conditions. Oxalate reacts very slowly at room temperature so the solutions are titrated hot to make the procedure practical. The unbalanced redox reaction is shown below.

$$MnO_4^- + C_2O_4^{2-} \rightarrow Mn^{2+} + CO_2 \text{ (acidic solution)}$$

Based on this reaction above, a potassium permanganate solution can be standardized against a sample of potassium oxalate; therefore the exact normality* (eq/L) of the permanganate solution can be determined. This is very useful because now the standardized permanganate solution can be used to find the concentrations of iron (II) in an unknown solution. The unbalanced redox reaction for the determination of an unknown iron (II) concentration is shown below.

$$MnO_4^- + Fe^{2+} \rightarrow Mn^{2+} + Fe^{3+} \text{ (acidic solution)}$$

This lab will be split up into two parts: standardizing the potassium permanganate solution and determining the concentration of Fe(II) in an unknown solution. *This lab utilizes a chemical term, normality, to describe the concentration of a solution. *Normality is a useful scale to describe the transfer of electrons in a redox reaction*. The units of normality are # of equivalents/liter, which for this lab can be related to the # of electrons/liter.

Pre-lab questions:

1. Using the half-reaction method, write a balanced redox equation for the reaction of permanganate with oxalate in an acidic solution.
2. How many electrons lost or gained by each half reaction?
3. For the reaction of $KMnO_4$ with $K_2C_2O_4$, use Nernst equation to find the equilibrium constant of this reaction?

Table 8.1: Chemicals and Supplies

Chemicals	Supplies
Distilled water	Burette
$KMnO_4$	500 mL Erlenmeyer flask(1)
$K_2C_2O_4$	250 mL Erlenmeyer flask (1)
6N H_2SO_4 (prepare by TA)	100 mL Graduated cylinder (1)
6 N H_3PO_4 (prepare by TA)	Stirring rod
Oxalic acid (for cleaning), (prepare by TA)	Thermometer
Fe (II) solution (prepare by TA as an unknown)	Hot plate

Procedure:

Part I: Prepare and Standardize $KMnO_4$ Stock Solution

1. Weigh approximately 0.1 g KMnO4 crystals using the weighing boat and transfer the crystals to a 500mL Erlenmeyer flask.

2. Use the graduated cylinder and measure 350 mL of distilled water and add it to the flask in step-1.

3. Heat the solution with occasional swirling to dissolve the $KMnO_4$ crystals.

   Note: **Do not boil the solution. This may take about 30 minutes. Allow the solution to cool.**

4. Use another weighing boat and weigh approximately 0.05 g of potassium oxalate ($K_2C_2O_4 \bullet H_2O$). Record the exact mass to 2 decimal places. Transfer the sample to a 250 mL Erlenmeyer flask.

5. Rinse and fill the burette with the $KMnO_4$ solution.

6. Add 50 mL of distilled water and 5 mL of 6 N H_2SO_4 to the oxalate sample in the Erlenmeyer flask. Swirl to dissolve the solids.

7. Heat the acidified oxalate solution to about 85°C. Do not boil the solution. Record the initial burette reading.

8. Titrate the hot oxalate solution with the $KMnO_4$ solution until the appearance of a very faint pink color. Record the final burette reading in table 8.2.

   Note: Discard the titration mixture in the appropriate waste container in the fume hood that prepared by the TA.

9. Repeat the titration with a new sample of oxalate for 2 more trials.

   Note: **An oxalic acid solution may be used to wash the burette and the titration flask if a brown stain remains in the glassware.**

Part II: Quantifying Fe^{2+} in unknown solutions

1. Pipet a 25 mL sample of the unknown Fe (II) solution into a 250 mL Erlenmeyer flask.
2. Add 50 mL of distilled water and 12 mL of 6 N H_3PO_4 into the flask.
3. Fill a burette with the standard $KMnO_4$ solution and record the initial burette reading.
4. Titrate the sample with the standard $KMnO_4$ to a faint pink end-point and record the final burette reading. Record the final volume of $KMnO_4$.

   Note: **Discard the Fe solution in the appropriate waste container that prepared by the TA.**

5. Repeat the titration with a new sample of the unknown Fe (II) solution for 2 more trials.

   Note: **Discard the purple permanganate solution in the appropriate waste container in the fume hood.**

Report Sheet of Exp. 8: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the table below

Table 8.2: collecting data for the reaction between $KMnO_4$ and $K_2C_2O_4$

	Initial volume (V_i) of $KMnO_4$	Final volume (V_f) of $KMnO_4$	$\Delta V = (V_f - V_i)$	Stock solution Normality of $KMnO_4$	Normality of standard $KMnO_4$
Trial 1					
Trial 2					
Trial 3					
Average	--------	-------	--------		

Table 8.3: collecting data for the reaction between $KMnO_4$ and F^{2+}

	Initial volume (V_i) of $KMnO_4$	Final volume (V_f) of $KMnO_4$	$\Delta V = (V_f - V_i)$	Standard solution Normality of $KMnO_4$	Normality of Fe^{2+} used
Trial 1					
Trial 2					
Trial 3					
Average	--------	-------			

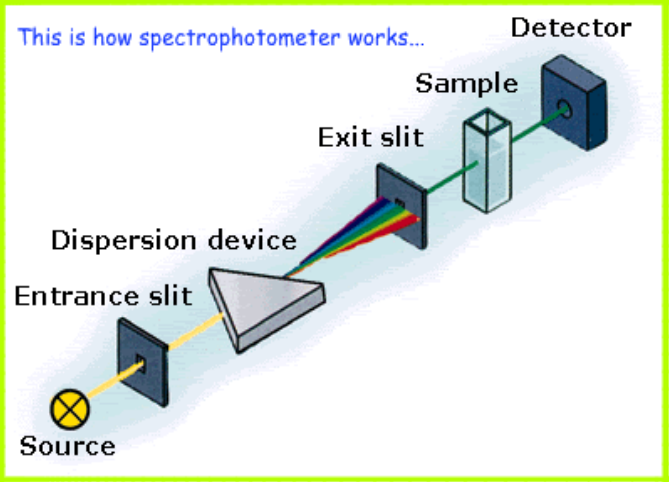
Part B: Answer the following questions:

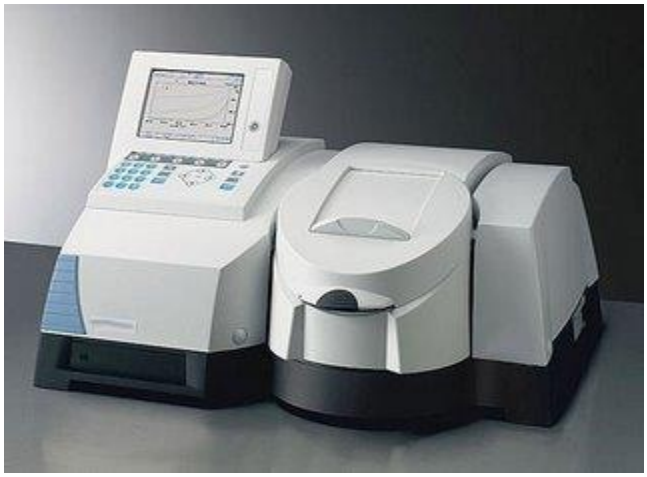
1. Using the half-reaction method, write a balanced redox equation for the reaction of permanganate with oxalate in an acidic solution.

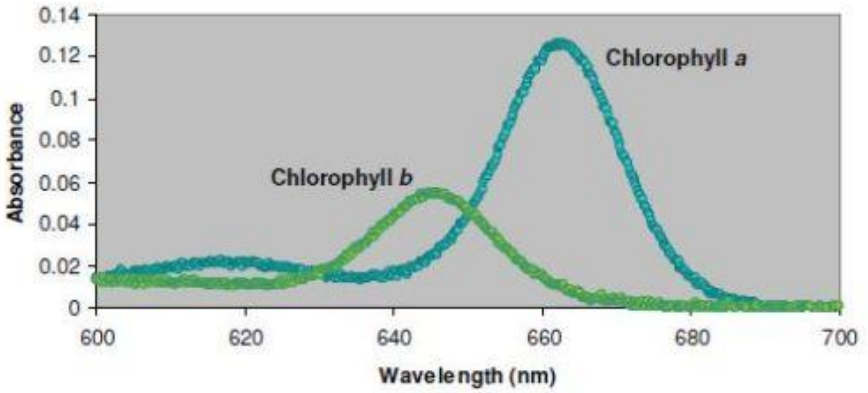
2. Using the half-reaction method, write a balanced redox equation for the reaction of permanganate with Fe^{2+} in an acidic solution.

3. Calculate:
   a. the normality of $KMnO_4$ stock solution
   b. The normality of standard $KMnO_4$ solution after titration,
   c. The average normality of standard $KMnO_4$.
   d. The normality of Fe^{2+} sample after titration,
   e. The average normality of standard Fe^{2+}.
   f. The molarity of Fe^{2+}.

Experiment 9: Photometric Determination of Equilibrium Constant







Objectives

1. This Experiment introduces the concept of equilibrium using the formation of a complex ion $Fe(SCN)_x^{(3-x)}$.
2. By determining the exact concentration of each species in equilibrium, the equilibrium constant, K, can derived and it can then best describe the direction of the reaction.
3. Have hands on using spectrophotometric technique.
4. Create a calibration curve and determine the concentration of $Fe(SCN)_x^{(3-x}$ by using Beer's Law, $A = \varepsilon bc$, which then used to derived the equilibrium constant, K, of the other species in the reaction.
5. Use Microsoft Excel to simplify the calculations.

Introduction

The use of light to measure chemical concentrations is called spectrophotometry. When you pass light through a sample, a certain amount of light is absorbed by the molecules in solution, and the rest passes through. In fact, materials have specific color due to certain wavelengths or colors of the visible spectrum that are being absorbed by the molecules in the material. The color you attribute to the material is actually all the remaining reflected light. If we measure the amount of light that is going into the sample and we measure the amount of light that comes out, we can determine to what extent the sample is absorbing the light.

In this experiment the equilibrium concentration of a complex ion is to be determined by use of absorption spectroscopy. The first part of the experiment, however, requires the determination of extinction coefficient, ε, The Beers-Lambert relationship between concentration and absorption is used to create a calibration graph. As long as one concentration in the reaction remains constant, the graph will then be (hopefully) linear with a slope equal to ε.

In order to generate a calibration curve, the absorbance of series of solutions with different concentrations of the colored species is measured at specific wavelength. A plot of absorbance (y-axis) versus [KSCN] (x-axis) is created a linear graph corresponding to the equation A = εbc, where the extinction coefficient is equal to the slope. This extinction coefficient is then used in a later series of reactions to calculate the actual concentration of the colored complex.

The reaction below poses some interesting challenges because the exact chemical formula of the product is unknown.

$$Fe^{3+}(aq) + xSCN^{-}(aq) \rightleftharpoons Fe(SCN)_x^{(3-x)}(aq)$$

The subscripts and coefficients represented with "x" shown in the reaction above could be 1, 2, or 3, yielding three different balanced reactions, respectively:

$$Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons Fe(SCN)^{2+}(aq) \quad \text{when } x = 1$$

or

$$Fe^{3+}(aq) + 2SCN^{-}(aq) \rightleftharpoons Fe(SCN)_2^{+}(aq) \quad \text{when } x = 2$$

or

$$Fe^{3+}(aq) + 3SCN^{-}(aq) \rightleftharpoons Fe(SCN)_3(aq) \quad \text{when } x = 3$$

In order to determine the equilibrium constant, the equilibrium concentrations of all the products and reactants must be determined several times starting with different initial concentrations of reactants. The calculations simply must be completed 3 times; once for each possible x value. By calculating the average and standard deviations of the equilibrium constant for each run, the value that produces the most consistent value of K (smallest standard deviation) will be the correct value to assume for the reaction.

Pre-lab questions:

1. You would like to determine the equilibrium constant for the binding of a ligand to a metal ion:

$$M^{3+} + L^{-} \rightleftharpoons ML^{2+}$$

$K_{eq} = \frac{[ML2+]}{[M3+][L-]}$, where K_{eq} is the equilibrium constant for this reaction

The ML^{2+} complex has a unique absorbance in the visible region, so its concentration at equilibrium can be determined by measuring the absorbance of the solution at an appropriate wavelength. The concentration of M^{3+} and L^{-} can be determined from their initial concentrations (refer to lecture). The following experiment is carried out first in order to determine the extinction coefficient, ε, of the complex at the wavelength chosen:

Solution	0.200 M Metal ion (mL)	0.1 M HNO_3 (mL)	0.0011 M Ligand ion (mL)	Solution concentration (M)	Absorbance	Extinction coefficient (M^{-1} cm^{-1})
A	5.0	4.0	1.0		0.113	
B	5.0	3.0	2.0		0.227	
C	5.0	2.0	3.0		0.337	
D	5.0	1.0	4.0		0.431	
E	5.0	0.0	5.0		0.562	

a. Calculate the complex concentration in each solution and then its extinction coefficient
b. Plot the absorbance versus the solution concentration and determine the slope and the intercept of the resulting line.

2. A student does an experiment to determine the equilibrium constant for the same reaction that you will study, but at a higher temperature.

$$Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$$

The student mixes 5.00 mL of 2.00×10^{-3} M $Fe(NO_3)_3$ solution with 5.00 mL of 2.00×10^{-3} M KSCN solution, heats the mixture, and finds that the equilibrium concentration of $FeSCN^{2+}$ in the mixture is 5.00×10^{-5} M.
Calculate the equilibrium constant for the reaction under the conditions in this experiment.

Table 9.1: Chemicals and Supplies

Chemicals	Supplies
$Fe(NO_3)_3$	Spectrophotometer (double beam)
KSCN	Cuvette
0.5 M HNO_3 (prepare by TA)	100 mL volumetric flask (1)
	50 mL volumetric flask (2)
	25 mL volumetric flask (5)
	10 mL volumetric flask (5)
	10 mL graduated pipette (3)
	100 mL or 50 mL beakers (2)

Procedure:

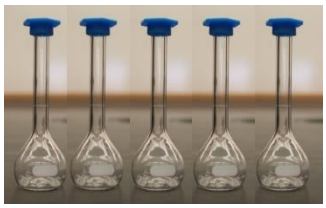
Part I: Preparing Solutions:

1. Use the dilution equation to prepare 0.1 M HNO_3 in 100 mL volumetric flask and label your flask.
2. Prepare 0.200 M $Fe(NO_3)_3$ in a 50 mL volumetric flask.

   Note: **add 5 mL of 0.1 M HNO_3 to the $Fe(NO_3)_3$ volumetric flask to acidify the solution**.
3. Prepare 0.0011 M KSCN in a 50 mL volumetric flask.

Part II: Using the Solutions to Determine the Extinction Coefficient:

1. Label 100 mL beaker as $Fe(NO_3)_3$ and pour the 50 mL volumetric flask that contain $Fe(NO_3)_3$ in it.
2. Label another 100 mL beaker as KSCN and pour the 50 mL volumetric flask that contains KSCN in it.
3. Line up 25 mL volumetric flask on your bench as showing in the picture below and label them from A – E.



4. Using the graduated pipette (one for each solution in the beakers) create the solutions A – E as described in the table 9.2 below:

Table 9.2: one reactant is changing its concentration while the other remains constant

Solution	0.200 M $Fe(NO_3)_3$ (mL)	0.0011 M KSCN (mL)	0.1 M HNO_3 (mL)	Absorbance
A	5.0	1.0	19.0	
B	5.0	2.0	18.0	
C	5.0	3.0	17.0	
D	5.0	4.0	16.0	
E	5.0	5.0	15.0	

Note: **Be very careful not to contaminate your stock solutions in the beakers. If a rust color should form in the pipette it is contaminated. Clean it and remake the solution you were working on.**

Part III: Measuring the Absorbance

1. Take the cuvette from the TA. Make sure it is clean and dry.
2. Set the spectrophotometer to wavelength 480 nm.
3. Create a blank of 0.200 M $Fe(NO_3)_3$.

4. Rinse the cuvette with the blank.

5. Fill the two cuvettes with the blank and zero the spectrometer.

6. Rinse one of the cuvette with the first solution that you prepared in one of the 25 mL volumetric flask (solution A)

7. Fill the cuvette in step 6 with solution A.

8. Measure and record the absorbance of solution A in your notebook.

9. Repeat steps 6-8 for the rest of the solutions, solution B to solution E.

10. Clean your mess.

Part IV: Making Solutions to Determine the Equilibrium Constant

1. Again, use the solutions in steps 1-2 (part II).

2. Line up 10 mL volumetric flask on your bench as showing in the picture below and label them from F – J.

3. Using the graduated pipette (one for each solution in the beakers) create the solutions F – J as described in the table below:

Table 9.3: different concentrations of the reactants

Solution	0.0023 M $Fe(NO_3)_3$ (mL)	0.0011 M KSCN (mL)	Absorbance
F	3.0	7.0	
G	4.0	6.0	
H	5.0	5.0	
I	6.0	4.0	
J	7.0	3.0	

Note: **Be very careful not to contaminate your stock solutions in the beakers. If a rust color should form in the pipette it is contaminated. Clean it and remake the solution you were working on.**

4. Repeat part IV for measuring the absorbance of solutions F – J.

Report Sheet-1 of Exp. 9: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the table below

Table 9.4: collecting data from part III

Solution	0.0023 M $Fe(NO_3)_3$ (mL)	0.0011 M KSCN (mL)	0.1 M HNO_3 (mL)	Absorbance	Initial $[SCN^-]$ M
A	5.0	1.0	19.0		
B	5.0	2.0	18.0		
C	5.0	3.0	17.0		
D	5.0	4.0	16.0		
E	5.0	5.0	15.0		

Table 9.5: collecting data from calculations and measurement in part IV

Solution	0.0023 M $Fe(NO_3)_3$ (mL)	0.0011 M KSCN (mL)	Initial $[Fe^{3+}]$ M	Initial $[SCN^-]$ M	Absorb-Ance (A)	$[Fe(SCN)_x^{(3-x)}] = A/\varepsilon b$	$[Fe^{3+}]eq$	$[SCN^-]eq$
F	3.0	7.0						
G	4.0	6.0						
H	5.0	5.0						
I	6.0	4.0						
J	7.0	3.0						

Table 9.6: Finding Keq using ICE table when x = 1

$Fe^{3+}(aq) + x\ SCN^-(aq) \rightleftharpoons FeSCN^{2+}(aq)$

	$[Fe^{3+}]$	X $[SCN^-]$	$[Fe(SCN)_x]^{(3-x)}$
I			
C			
E			

Note: you repeat designing this table when x = 2 and when x = 3.

Report Sheet-2 of Exp. 9: (Attach any extra paper to this sheet)

Table 9.7: Collect data when x =1

Solution	Keq	Av. Keq	(Keq – Av. Keq)	(Keq – Av. Keq)^2
F		(Sum Keq) /5		
G		(Sum Keq) /5		
H		(Sum Keq) /5		
I		(Sum Keq) /5		
J		(Sum Keq) /5		
	Sum Keq=	(Sum Keq) /5		Sum (Keq – Av. Keq)^2=

Table 9.8: Collect data when x =2

Solution	Keq	Av. Keq	(Keq – Av. Keq)	(Keq – Av. Keq)^2
F		(Sum Keq) /5		
G		(Sum Keq) /5		
H		(Sum Keq) /5		
I		(Sum Keq) /5		
J		(Sum Keq) /5		
	Sum Keq=	(Sum Keq) /5		Sum (Keq – Av. Keq)^2=

Table 9.9: Collect data when x =3

Solution	Keq	Av. Keq	(Keq – Av. Keq)	(Keq – Av. Keq)^2
F		(Sum Keq) /5		
G		(Sum Keq) /5		
H		(Sum Keq) /5		
I		(Sum Keq) /5		
J		(Sum Keq) /5		
	Sum Keq=	(Sum Keq) /5		Sum (Keq – Av. Keq)^2=

Report Sheet-3 of Exp. 9: (Attach any extra paper to this sheet)

Part B: Calculation the following:

1. Do the following calculations from part III measurements:
   Initial [SCN^-] = $(volume\ SCN^- \times [SCN^-])/(total\ volume\ of\ solution)$

2. Plot the absorbance (y-axis) versus [KSCN] (x-axis) and create a linear graph corresponding to the equation A = εbc, where the extinction coefficient, ε, is equal to the slope. (You should use excel sheet for this task)

3. For Part IV, For equilibrium data (solutions F – J), consider the following chemical reaction and equilibrium expression: Fe^{3+} (aq) + xSCN^- (aq) ⇌ $Fe(SCN)_x^{(3-x)}$ (aq)

   Keq = $[Fe(SCN)_x^{(3-x)}]/[Fe^{3+}][SCN^-]^x$, where x is 1, 2, or 3.

4. In order to determine the value of x, complete table 9.5 **for each x** by calculating the following

   Initial [SCN^-] = $(volume\ SCN^- \times [SCN^-])/(total\ volume\ of\ solution)$ ---------- (1)
   Initial [Fe^{3+}] = $(volume\ Fe^{3+} \times [Fe^{3+}])/(total\ volume\ of\ solution)$ ---------- (2)
   $[Fe(SCN)_x^{(3-x)}]$ can be found using Beer's equation: A = εbc ---------- (3)

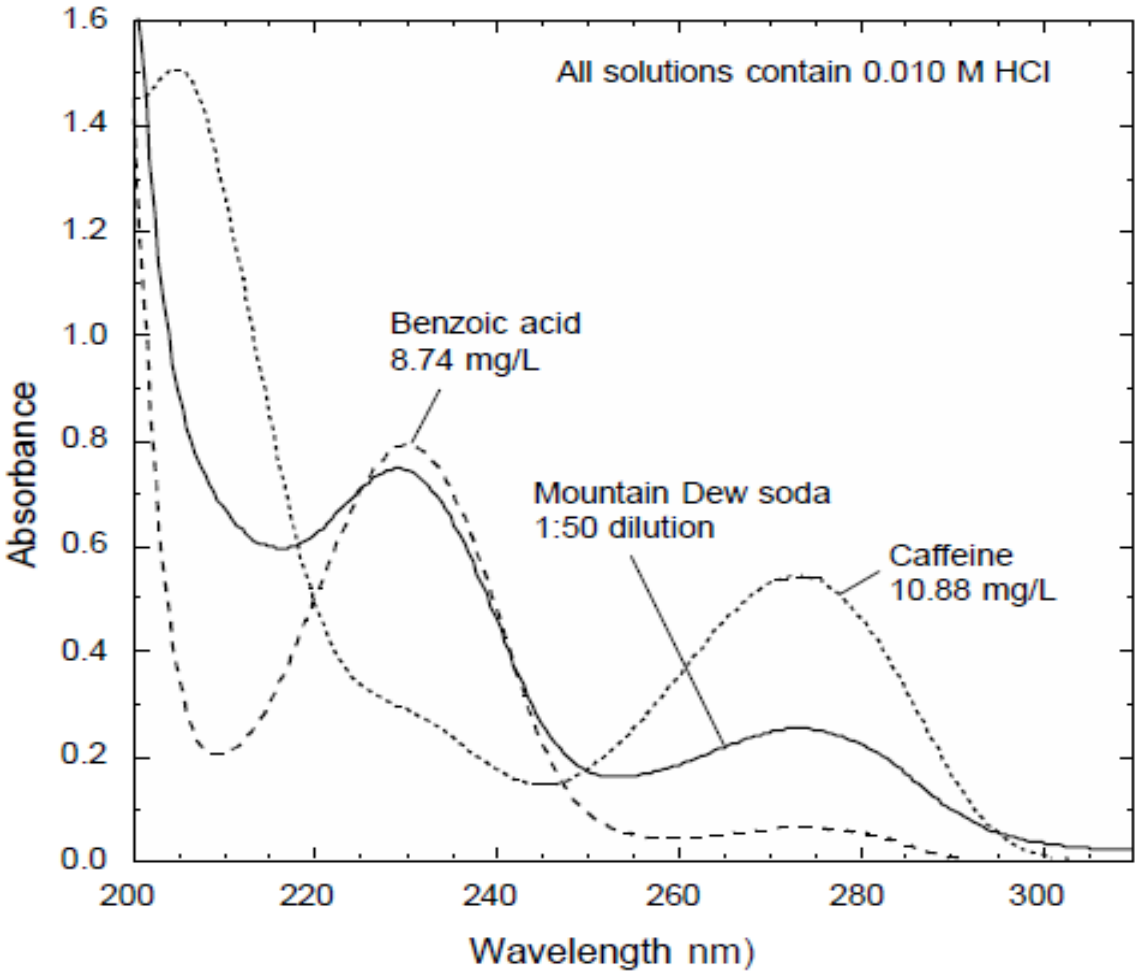
   $[Fe^{3+}]_{eq}$ = [Fe^{3+}] Init – $[Fe(SCN)_x^{(3-x)}]$ ---------- (4)
   $[SCN^-]_{eq}$ = x [SCN^-] Init – $[Fe(SCN)_x^{(3-x)}]$ ---------- (5)
   Keq = $[Fe(SCN)_x^{(3-x)}]/[Fe^{3+}][SCN^-]^x$ ---------- (6)

5. Use equation (6) and you tabulate your data using table 9.6:

6. Use excel sheet and create tables 9.7, 9.8 and 9.9 for each x value and calculate the average of Keq and the standard deviation in Keq.
   Standard deviation = $\sqrt{variance}$
   Variance = $\frac{\sum(xi-\bar{X})2}{n-1}$, where x_i is the Keq value for each run, $\bar{X}$ is the mean.

   Note: **the value that produces the most consistent value of K (smallest standard deviation) will be the correct value to assume for the reaction.**

Experiment 10: Spectrophotometric Analysis of a Mixture: Caffeine and Benzoic Acid in a Soft Drink

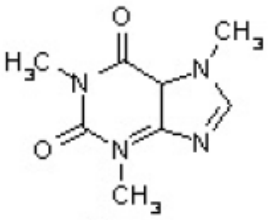


Objective:

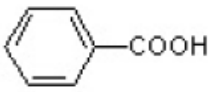
1. Using ultraviolet absorbance to measure two major species in soft-drinks. Caffeine is added as a stimulant and sodium benzoate is a preservative.
2. Use Lambert Beer's law to determine concentration of a substance exist in a mixture.

Introduction:

Two major chemical species found in soft drinks and 'energy' drinks are caffeine, added as a stimulant, and sodium benzoate, added as a preservative. The structures of these species are shown below:



Caffiene



Benzoic Acid

As you can see both compounds have an aromatic ring system, so you might guess that they should absorb light energy in the ultraviolet range. Caffeine has UV absorbance at about 205 and 275 nm, while benzoic acid absorbs at about 230 nm. These absorbances are just far enough apart that we can determine the concentrations of these two species simultaneously using their UV absorption.

In doing this analysis we have to avoid both drinks that have a dark color and those with artificial sweeteners, because these compounds have additional absorbances in the UV range. Even our lighter colored, sugar sweetened drinks still contain some interfering compounds, so our results will not be absolutely correct.

All solutions will contain 0.010 M HCl, so the sodium benzoate is protonated to make benzoic acid. Caffeine has no appreciable basicity, so it is neutral at pH 2.

The procedure we describe includes the construction of calibration curves. The experiment could be shortened by recording just one spectrum of caffeine (20 mg/L) and one of benzoic acid (10 mg/L) and assuming that Beer's law is obeyed.

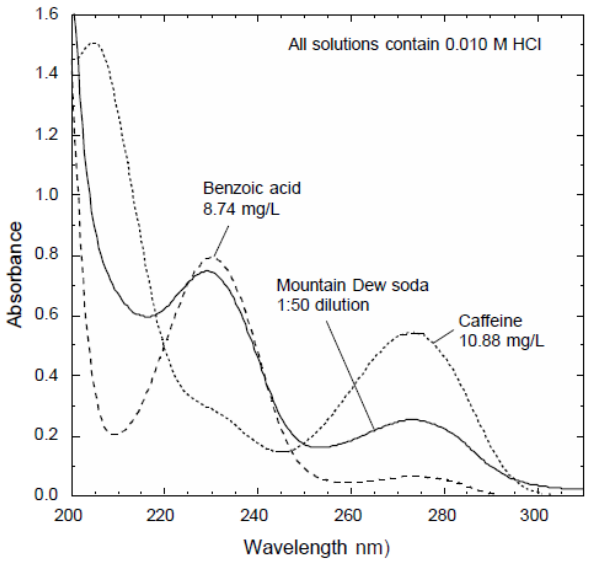


Figure 1. Ultraviolet absorption of benzoic acid, caffeine, and a 1:50 dilution of Mountain Dew soft drink. All solutions contain 0.010 M HCl.

Table 10.1: Chemicals and Supplies

Chemicals	Supplies
Soft drink (one can for each group)	Spectrophotometer (double beam)
100 mg/L Benzoic acid (prepare by TA)	Cuvette
200 mg/L Caffeine (prepare by TA)	25 mL volumetric flask (13)
0.1 M HCl (prepare by TA)	10 mL pipette (2)
	100 mL beaker (3)
	Filter paper (provide when it requested)
	Funnel (provide when it requested)

Procedure:

Part I: Calibration curve for standard solutions

1. Label five volumetric flask of the size (25 mL) as: A, B, C, D, E.

2. Bring a clean beaker and label it (benzoic acid), then from the 1L container measure about 50 mL of benzoin acid solution and add it into your beaker.

   Note: when you are done from step 2, take your beaker and return back to your place to continue your work.

3. Prepare benzoic acid solutions using the labeled volumetric flask as shown in the below table.

   Table 10.2: preparing standard solution of benzoic acid

A	B	C	D	E
0.5 mL	1 mL	1.5 mL	2 mL	2.5 mL

4. Add 2.5 mL of HCl to each volumetric flask then add distilled water until you reach to the mark and leave them a side.

   Note: if you crossed the mark, then you have to discard your solution and make a new one.

5. Label another five volumetric flasks of the size (25 mL) as F, G, H, I, J.

6. Repeat step 2-4, but using Caffeine solution.

   Note: you are going to use the same table 10.2 but the label of the flasks are different.

7. Prepare a blank by adding 2 ml of distilled water in a volumetric flask labeled as blank, then add 2.5 mL of HCl to the container and continue adding DW to the marks.

8. Use the spectrophotometer to measure the absorbance of the blank first then all solutions A-J at 273 nm once and at 230 nm at another time. Record your measurements in table 10.3 and 10.4.

Part II: Measure the soft drink

1. Warm ~20 mL of soft drink in a beaker on a hot plate to expel CO_2.

   Note: If you noticed and particles in your soft drink then filter the warm liquid through filter paper to remove any particles.

2. After cooling to room temperature, pipet 1.00 mL into a 25-mL volumetric flask. Add 2.5 mL of 0.1 M HCl and dilute to the mark.

3. In another clean volumetric flask, pipette 0.5 mL of the soft drink and transfer it to the volumetric flask then add 2.5 mL of 0.1 M HCl and dilute to the mark.

4. Use the spectrophotometer to measure the absorbance of the two solutions of the soft drink at 273 nm once and at 230 nm at another time and record your measurement in table 10.5.

Report Sheet-1 of Exp. 10: (Attach any extra paper to this sheet)

Group Names:

Part A: Fill the table below

Table 10.3: collecting data from part I

Solution	Concentration	Absorbance at 273 nm	Absorbance at 230 nm
A			
B			
C			
D			
E			

Table 10.4: collecting data from part I

Solution	Concentration	Absorbance at 273 nm	Absorbance at 230 nm
F			
G			
H			
I			
J			

Table 10.5: collecting data from part II

Soft Drink	Absorbance at 273 nm	Absorbance at 230 nm
1		
2		

Report Sheet-2 of Exp. 11: (Attach any extra paper to this sheet)

Part B: Do the following:

1. Calculate the concentration of solution A-E, use the dilution equation for this purpose.

2. Use the excel sheet to graph the concentration against the absorbance. The slope will represent the molar absorptivity at each wavelength.

   Note: you should generate 4 graphs and as a result you should have 4 molar absorptivity.

3. For the first solution of the soft drink, use the equation to find the concentration of caffeine and benzoic acid in soft drink:

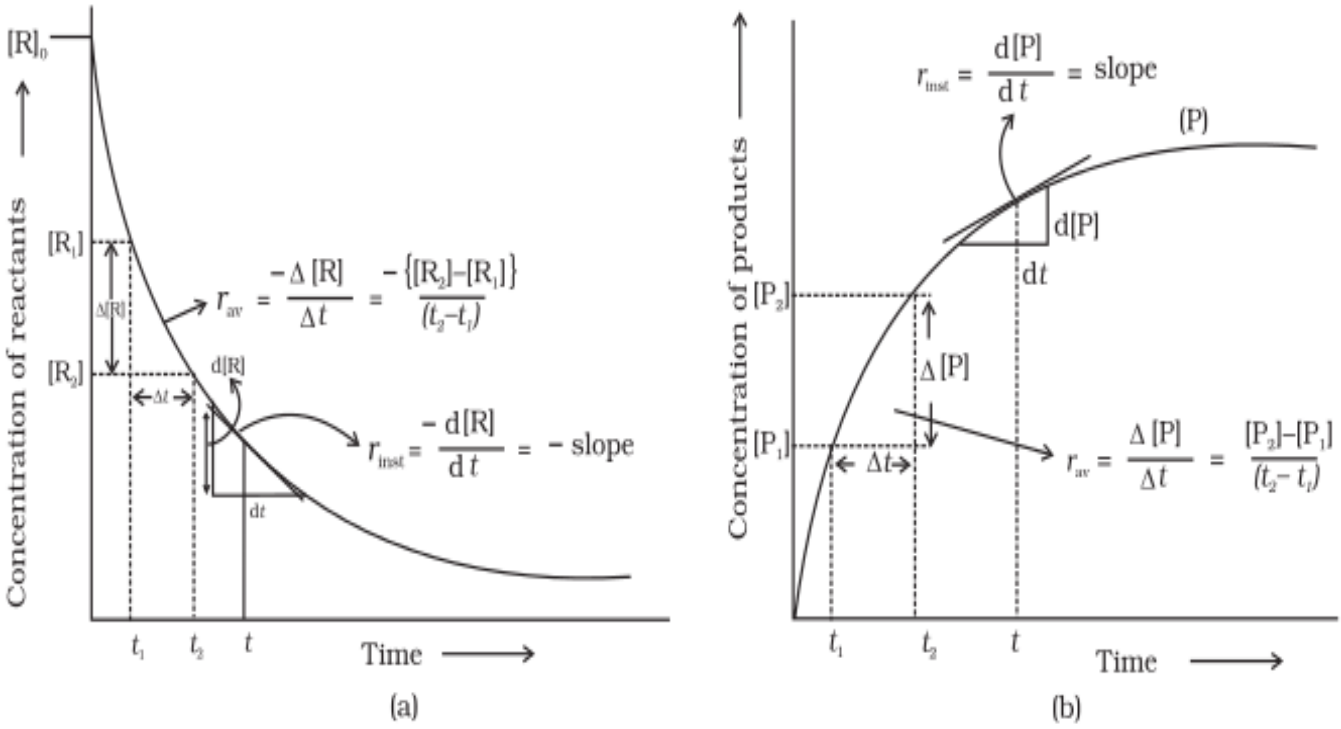
   $A_{\lambda 1} = \varepsilon_{Caff}\, b\, C_{Caff} + \varepsilon_{benz}\, b\, C_{benz}$

   $A_{\lambda 2} = \varepsilon_{Caff}\, b\, C_{Caff} + \varepsilon_{benz}\, b\, C_{benz}$

4. For the second solution of the soft drink, repeat the calculations in step 3.

   Note the absorbance of the second solution is different than the first, therefore, you should collect a different results.

Experiment 11: Kinetics Iodine Clock



Objectives

1. Get experience on preparing certain concentrations of different solutions.
2. Determine the orders of the reactants.
3. Understand the factors affect the rate of the reaction

Introduction

The study of rates of chemical reactions is referred to as **chemical kinetics**. The study of kinetic is very important for manufacturers. In the production of various chemicals, it is vital to know the rate at which a product is both produced and broken down. For example, in the production of steel, the proportions of iron and carbon are varied based on the intended use of the steel. The cooling of the molten steel (known as austempering) is controlled to allow a desired crystal structure to develop; these crystal structures have a specific names and properties. When bainite (a type of iron) forms in austenite (a mixture of iron and carbon), the content of the austenite increases as the transformation occurs. If the austempering stage is too short, martensite may form in the residual austenite. If the reaction takes too long, carbide precipitation may occurs. Both phenomena are detrimental to the mechanical properties of the steel that is being produced. It is therefore necessary to know how long austempering treatment should take.

Another commonplace example is the expiration dates or shelf lives of foods and drugs. Knowledge of the kinetics of the chemical reactions that break down certain chemicals present in food or drug products can be used to determine how potent or unsafe a food or a drug may be after a certain period. The same knowledge of reaction rates for specific chemicals can also be used to determine the dosage of a drug. For example, if the drug is slow to react within the body, the dosage can be larger because it is naturally "time-released" and thus won't hit the system all at once. If the reaction rate is very fast, too high of a dose might kill the patient. A good knowledge of reaction rates is therefore lifesaving and its of importance to the creation of miscellaneous chemical products.

In studying the chemical kinetics of a system, we need to know what the rate means, how to determine the reaction rate experimentally, how the temperature and concentration affects the reaction rate and a detailed pathway taken by the atoms as the reaction proceeds, also known as the reaction mechanism.

Fundamental observation in reaction kinetics is of a reactant being consumed or the products being formed in a given time interval. Concentration data may then be used to calculate the rate of reaction for a given experiment according to the following equations:

Rate of Reaction = Amount of Reactant Consumed/Time of Observation = - d[Reactant]/dt

Or Rate of Reaction = Amount of Product Created/Time of Observation = d[Product]/dt

In order to measure a reaction rate, there must be some method of determining the concentration of reactants or products at the beginning and at the end of a time period. For this experiment, we will vary the initial concentration of a reactant, and measure the time until the reactant is completely consumed. For most reactions, the rate decreases continuously as you progress in the reaction. This is due to a decrease in the concentrations of kinetically significant reactants.

Pre-lab Questions

1. For KI, $Na_2S_2O_3$, and $(NH_4)_2S_2O_8$ calculate the initial concentration (M) based on the table of solution concentrations and volumes given in Table 4.4.

Table 11.1: Finding Initial concentration

Run #	[KI] M	$[Na_2S_2O_3]$ M	$[(NH_4)_2S_2O_8]$ M	Time
1				
2				
3				
4				
5				

2. A first-order chemical reaction has a rate constant of $9.1\times10\ S^{-1}$ at 12˚C and a rate constant of $4.1\times10^2\ S^{-1}$ at 36˚C. Calculate the activation energy, E_a, for the reaction.

3. Use the data in table 4.2 to determine:
   a. The orders of the reaction (m and n) for reactant A and B.
   b. The rate constant (k) for the reaction below.

$$A_{(aq)} + B_{(aq)} \longrightarrow \text{Products}$$

Table 11.2: Initial concentration of the reactants A and B

Run #	[A], M	[B], M	Rate of Loss of A, M/s
1	0.25	0.25	9.0×10-3
2	0.25	0.50	3.6×10-2
3	0.50	0.25	1.8×10-2

Table 11.3: Chemicals and Supplies

Chemicals	Supplies
Ice cubes	250mL Volumetric flask (1)
0.2 M Potassium iodide	100mL Volumetric flask (2)
0.050 M Ammonium peroxydisulfate, $(NH_4)_2S_2O_8$	25mL Volumetric flask (1)
0.00175 M Sodium thiosulfate, $Na_2S_2O_3$	50mL graduated cylinder (4)
Starch (prepare by TA)	500mL Erlenmeyer flask (2)
	Stopwatch (1)

Procedure

Part A: Prepare the following solutions

1 0.20 M KI in 250mL V.F

2 0.050 M $(NH_4)_2S_2O_8$ in 100mL V.F

3 0.00175 M $Na_2S_2O_3$ in 100mL V.F.

Part B: Finding the reactants' order and the rate constant

1 Use a clean 500 mL Erlenmeyer flask to combine the reagents for each run (1-5) listed in table 8.4 below.

Note: please clean the Erlenmeyer flask after each run.

2 Use a different graduated cylinder to measure each reagent. Label your cylinder to avoid mixing between them.

3 The reagents should be added one at a time into the Erlenmeyer flask in the order listed in table 11.4.

4 Use the stopwatch provided to you and be ready to start timing the reaction when the final reagent $(NH_4)_2S_2O_8$, is added.

5 Stop timing each reaction when the first appearance of blue color is seen. Record the elapsed time in table 11.5.

Table 11.4: Initial concentrations of reactants

Run #	KI 0.20M	$Na_2S_2O_3$ 0.00175 M	Water	Starch	$(NH_4)_2S_2O_8$ 0.050 M
1	30 mL	20 mL	0 mL	3 Drops	30 mL
2	20 mL	20 mL	10 mL	3 Drops	30 mL
3	10 mL	20 mL	20 mL	3 Drops	30 mL
4	30 mL	20 mL	10 mL	3 Drops	20 mL
5	30 mL	20 mL	20 mL	3 Drops	10 mL

Report Sheet of Exp. 11: (Attach any extra paper to this sheet)

Group Names:

Part A: Collecting Data

Table 11.5: Recording data collected from the procedure

Run #	[KI] M	[$Na_2S_2O_3$] M	[$(NH_4)_2S_2O_8$] M	Time (Sec)	Temp. (˚C)
1					
2					
3					
4					
5					

Part B: Do the following calculations

1. Find the reaction orders (m and n), this can be happened by using the method of the initial rates.
2. Calculate the average rate constant (from run 1-5)
3. Find the rate law.

Part C: Answer the following questions:

1. Why this experiment called iodine clock?
2. What is the job of $Na_2S_2O_3$?
3. Explain any weak or strength point you noticed while you are conducting the experiment?
4. In your Opinion, how we can update (improve) this experiment?

References

- Dillon S., McMurry F. (2012). Laboratory Manual, Florida State University with contributions College of Pennsylvania, Pearson Prentice Hall. (AUIS)

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- Experiment 3: Chemical Equilibrium and Le Chatelier's Principle, pdf https://www.smc.edu/AcademicPrograms/PhysicalSciences/Documents/Chemistry_10_Experiments/Ch10_Equilibrium.pdf

- Experiment 5: Preparing Buffers and Buffer Capacity Lab, Westminster College, pdf https://www.westminster.edu/about/community/sim/pdf/spreparingbuffersandbuffercapacity.pdf

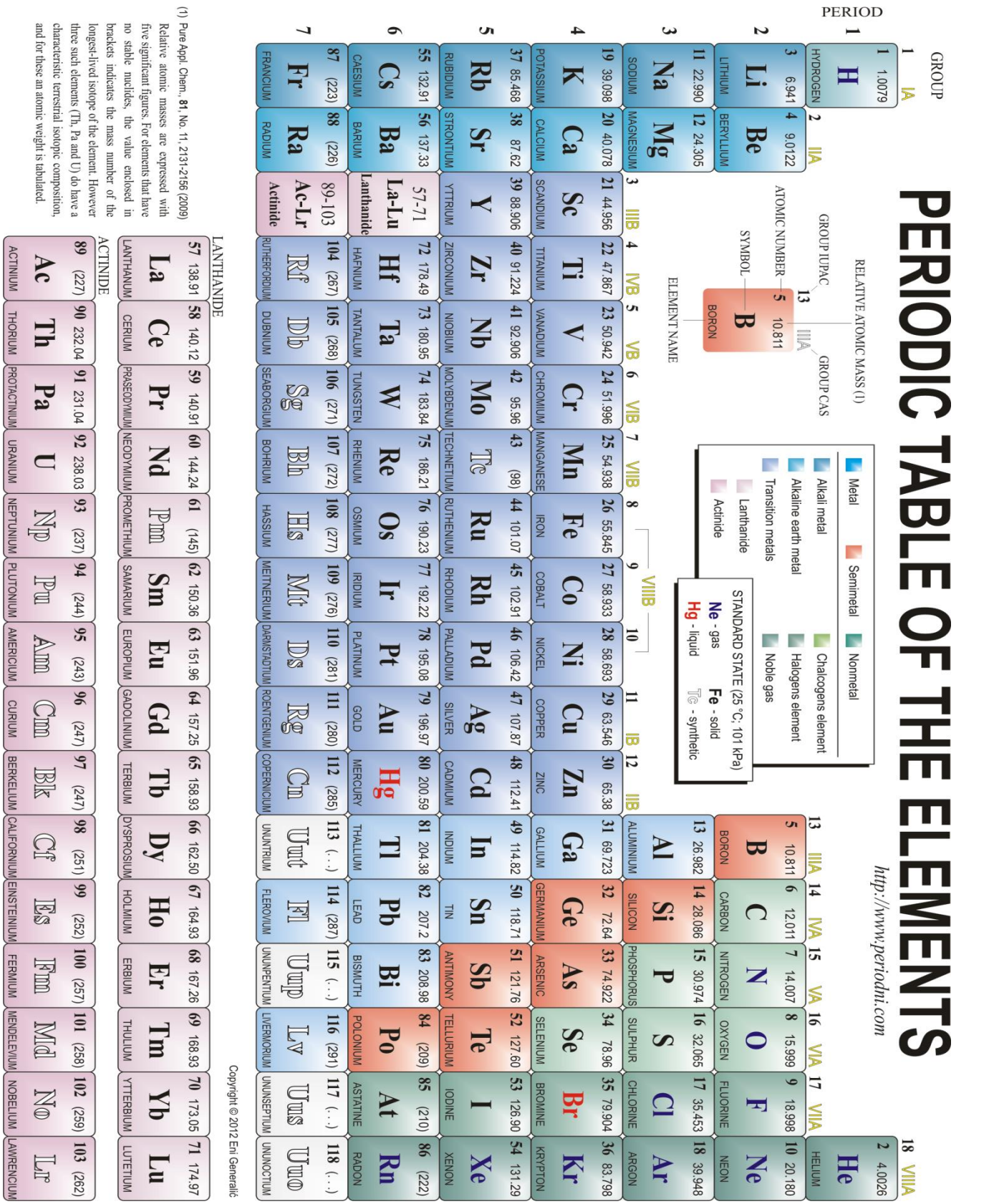
- Experiment 6: Determining the Concentration of Citric Acid in a Soft Drink Using Acid-Base Titration, pdf http://www.flavours.asia/uploads/7/9/8/9/7989988/titration.pdf

- Experiment 7: AP Chemistry Acid-Base Titration Lab, pdf http://www.kentchemistry.com/APlabs/Lab_AP_Titation.pdf

- Experiment 8: Redox Titration, pdf http://www.lahc.edu/classes/chemistry/arias/exp%208%20-%20redox.pdf

- Spectrophotometric Analysis of a Mixture: Caffeine and Benzoic Acid in a Soft Drink, pdf http://web.mnstate.edu/marasing/CHEM380/Labs/380PLABL/Spectrophotometric%20Analysis%20of%20a%20Mixture%20%20Caffeine%20and%20Benzoic%20Acid.pdf

Periodic Table of the Element



(1) Pure Appl. Chem., **81**, No. 11, 2131-2156 (2009)

Relative atomic masses are expressed with five significant figures. For elements that have no stable nuclides, the value enclosed in brackets indicates the mass number of the longest-lived isotope of the element. However three such elements (Th, Pa and U) do have a characteristic terrestrial isotopic composition, and for these an atomic weight is tabulated.