

# VC211 GENERAL CHEMISTRY LABORATORY

## Collaborative Investigations in Chemistry

### 2018-2019 Laboratory Manual

by

UM-SJTU JI & School of Chemistry and Chemical Engineering, SJTU

"This manual is based on significant modification of experiments published originally in lab manual by Konigsberg Kerner & Penner-Hahn, Hayden McNeil; Hands on Chemistry Laboratory Manual, 1st Ed., Jeffrey A. Paradis, Kristen Spotz, McGraw Hill Higher Education Press, 2006"



University of Michigan – Shanghai Jiao Tong University (UM-SJTU JI)

800 Dongchuan Road, Shanghai, China

200240 Email: [ting.sun@sjtu.edu.cn](mailto:ting.sun@sjtu.edu.cn)

Web: <http://umji.sjtu.edu.cn>

In order to protect the copyrights of any portion of this lab manual, no one is authorized to copy, photograph, or reproduce in any form, any portion without the written consent of the original author(s) of each of the sections. This manual is only for instructional internal purposes at the University of Michigan – Shanghai Jiao Tong University Joint Institute (UM-SJTU JI) and not for sale or distribution. *Despite substantial efforts to ensure the completeness & correctness of this manual, the author(s) cannot guarantee the accuracy of any of the information given herein, including but not limited to the resources & references, experimental illustrations, instructions, procedures, safety warnings and health protection instructions, safety guidelines, chemical disposal guidelines, or any portion of this manual. The users of this manual are urged to seek additional references to minimize errors, omissions, or any possible potential hazard that may be presented.*



## EMERGENCY CONTACT INFORMATION

**“Anytime you have an emergency, please call immediately your local police and the numbers below.**  
**You must immediately seek medical help from the nearest medical emergency facility or hospital.**  
**In case of fire notify immediately the nearest fire department and take actions to contain the fire or exit the place at once if not possible”**

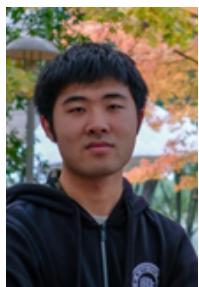
AGENCY	机构	CALL
CHEMISTRY BUILDING “A” SERVICES	化学楼	021-54742802
SJTU UNIVERSITY PUBLIC SAFETY	交大保卫处	021-54749110
CAMPUS EMERGENCY MEDICAL SERVICES	校医院急诊	021-54742400

**CALL BELOW ONLY DURING VC211 LAB SESSION OF SPRING 2019.**

**COURSE INSTRUCTOR: Dr. Ting Sun Telephone: 021-34206765-4391**

TA'S ASSIGNMENT SCHEDULE IS SUBJECT TO CHANGE. CHECK CANVAS FOR THE LATEST CHANGES.

SEC	TA NAME Pinyin	TA's phone	TA's email	LAB DAY: Chem Bldg. A	TA's Office Hour/ Location
1 7	Wu, Qirui 吴启瑞	15365100929	<a href="mailto:wuqirui@sjtu.edu.cn">wuqirui@sjtu.edu.cn</a>	08:00-11:00(W) 08:00-11:00 (F)	TBD
2 8	Huang, Pengyuan 黄鹏远	18621812875	<a href="mailto:1140833585hpy@sjtu.edu.cn">1140833585hpy@sjtu.edu.cn</a>	08:00-11:00(W) 08:00-11:00 (F)	TBD
3 10	Zheng, Siyi 郑思仪	18621851244	<a href="mailto:tang_c@sjtu.edu.cn">tang_c@sjtu.edu.cn</a>	18:00-21:00 (W) 13:00-16:00 (F)	TBD
4 12	Chi, Pengnan 迟朋南	18621810298	<a href="mailto:shawntcpn@sjtu.edu.cn">shawntcpn@sjtu.edu.cn</a>	18:00-21:00 (W) 18:00-21:00 (F)	TBD
5 11	Guo, Yunjie 郭蕴捷	18621957432	<a href="mailto:gyj990713@sjtu.edu.cn">gyj990713@sjtu.edu.cn</a>	18:00-21:00 (TH) 18:00-21:00 (F)	TBD
6 9	Zhao, Yuntian 赵云天	15801799167	<a href="mailto:clairez@sjtu.edu.cn">clairez@sjtu.edu.cn</a>	18:00-21:00 (TH) 13:00-16:00 (F)	TBD
	Zhao, Yiqing 赵漪青	13611793719	<a href="mailto:cissyy1999@sjtu.edu.cn">cissyy1999@sjtu.edu.cn</a>	TA Coordinator	TBD



Wu, Qirui  
吴启瑞



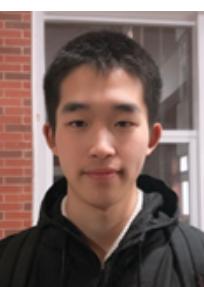
Huang, Pengyuan  
黄鹏远



Zheng, Siyi  
郑思仪



Chi, Pengnan  
迟朋南



Guo, Yunjie  
郭蕴捷



Zhao, Yuntian  
赵云天



Zhao, Yiqing  
赵漪青

## **IMPORTANT EMERGENCY & SAFETY NOTES:**

- 1. CONTACTS MAY CHANGE WITHOUT NOTICE. YOU MUST CONFIRM AND UPDATE THE CONTACT INFORMATION YOURSELF**
2. Laboratory location: Chemistry building A, room A224 or A226, SJTU, Minhang Campus. Check Canvas and JI undergraduate office announcements in case location changes.
3. Report any safety issues immediately to the TA, instructor and the Chemistry department.
4. **Safety and Laboratory Needs:** Students must adhere to the strict regulations with respect to laboratory safety, follow safety instructions posted by the chemistry building and the laboratory manual. Absolutely, at least the following must be adhered to inside the laboratory: no smoking, no drinking, no food product or consumption or eating, no shorts, no sandals, and you must wear laboratory safety goggles (glasses) at all times. Disposal of any unauthorized chemical waste into the environment or drain system is prohibited by-law. You must ensure your safety and the safety of others. **You must seek help if you face any safety issue, so please make sure you have in your possession the Emergency Contact Information.**

## **SAFETY ANNOUNCEMENT**

***"Substances used/handled during the term may carry health risks for certain students despite efforts to minimize any potential hazard. For example, certain students may have allergies related to some chemicals. If any student has particular health concerns, seek medical advice and decide whether to continue or drop the class. Refer to the more detailed safety instructions in Appendix (A) of this manual or as may be announced on JI Canvas online"***

The author(s) strongly urges all persons involved with chemical disposal to consult their chemical suppliers for detailed instructions for the disposal of individual chemicals. In addition, reviewing the government rules, regulations and literature are helpful. All persons are recommended to adhere to the strict government rules and regulations with respect to laboratory safety, health and environmental protection. Disposal of unauthorized chemical waste into the environment or drain system is prohibited by-laws. You are recommended to ensure safety and health standards for yourself and others. You should seek immediate help if you face any safety or health hazard issue, so please make sure you have in your possession a readily available emergency contact information of local police, fire department and the nearest qualified emergency health care facility.



## TABLE OF CONTENTS

	PAGE
EMERGENCY CONTACT INFORMATION .....	i
SAFETY ANNOUNCEMENT .....	ii
VC211 GENERAL CHEMISTRY LABORATORY SYLLABUS .....	v
EXPERIMENT E1: Chemistry of the Kitchen – Acids & Bases .....	E1: 1
EXPERIMENT E2: The Properties of Buffer: Resisting Change in a Turbulent World .....	E2: 1
EXPERIMENT E3: Spectrophotometric Analysis: Phosphates in Water .....	E3: 1
EXPERIMENT E4(I): Introduction to Kinetics: Factors that Affect the Rate of Reaction .....	E4(I): 1
EXPERIMENT E4(II): Determining the Rate Law: A Kinetics Study of Iodination of Acetone .....	E4(II): 1
EXPERIMENT E5: Precipitation and Water Purity .....	E5: 1
APPENDIX (A): LABORATORY INSTRUCTIONS TO TA'S .....	p1
PART I: GENERAL INSTRUCTIONS TO TA-s .....	p1
PART II: LABORATORY SAFETY INSTRUCTIONS .....	p5
PART III: GRADING, REPORTS, & PPT INSTRUCTIONS .....	p12
APPENDIX (B): ADDITIONAL REFERENCES .....	p25



## VC211 TYPICAL GENERAL CHEMISTRY LABORATORY SYLLABUS

**“Read & understand this syllabus thoroughly including safety warnings & instructions. If you violate the safety rules you may be dismissed from the lab. Please do not pollute & be responsible to yourself & others”**

**“Syllabus subject to change at any time, please confirm all schedules with JI undergraduate office or on Canvas”**

***Instructor: Dr. Ting Sun***

### **Course Description:**

Chemistry Laboratory VC211 is to foster critical thinking that allows students to design, perform, and interpret experiments. In addition, the student acquires technical skills that are required for further advancement in experimental sciences. An ability to collect and analyze data is developed, so the emphasis of the course is to provide a quantitative as well as a qualitative understanding of the basic concepts of chemistry. This is accomplished by demonstrating that chemical principles are derived from experimental data. The goal is to provide students both with a more accurate picture of the scientific process and also with skills that are relevant to solving real life problems. Chemistry VC211 is 1-credit and can be elected with, or following, General Chemistry.

In Chemistry Laboratory VC211, you will attend experimental lectures, do experiment and participate in discussion for 3 hours per week; the schedule is listed below. Students will do some experiments individually and finish some of the course work as a member of a team. Student groups each explore the same problem with each group using different reagents and/or conditions. Student groups address questions which require them to organize the laboratory data. Group answers are presented in discussion during laboratory hours.

The entire class is divided into **12 class sections** (1 lab/section) and a TA is assigned to lead each section. Each section will be divided into groups of 3-5 **students per group**. Each group conducts experiments jointly or individually as instructed. Starting the second week, each class section assembles in the laboratory area in the **Chemistry Building A (224 & 226)**, second floor, according to the schedule posted on CANVAS and as shown by this Syllabus. The entire students will meet once for 1 ½ hours during the **first week** for introduction lecture by Dr. Sun. There after students during the semester, will conduct a total of **5 assigned laboratory experiments, one experiment per week** (see class schedule at the end of this syllabus). Each team is required to give 10-15 minutes presentation on one of the experiments (E1-E5) that the TA will designate randomly. Students' PPT should include some photos documenting their experimental work.

**In addition students should give the TA, immediately before presenting their PPT, a short team efforts report on experimental design to analyze Ca in commercial products.** This year students will investigate on their own and propose experimental design and analysis of Ca content in common commercial products such as food, consumable products, or pharmaceutical vitamins. The students will rely on their work experience with experiments 1 & 3. The report should include abstract, introduction, theory, experimental setup, procedure, what data to measure (in suppliers supplementary products), & expected results. This design report should be condensed & similar to the depicted reports of any of their experiments and no more than 5 pages (or as posted by the announcements and instructions on Canvas or laboratory manual).

The **final exam** is scheduled on the 9<sup>th</sup> week (tentatively).

**Each student must check CANVAS very frequently to obtain the entire course materials, laboratory instructions, safety instructions, assigned experiments, announcements, sections assignment, grading, and detailed schedule & grouping.**

The format of the course is organized into **4 segments**:

**Segment 1, Week 1 Lecture:** The students will attend once the 1 ½ hour lecture, introduction to chemistry laboratory, by Dr. Sun, date **Wednesday (Feb. 27), 14:00-15:40** at **Shangyuan building Room A100**. Check CANVAS for confirmation of the schedule. After this lecture you will have no other scheduled meeting during the first week.

### **Segment 2: Week 2 - Week 6, Laboratory Work: Chemistry Building A**

- Students of each section will assemble on scheduled time once a week in Lab **Room A224**, and attend a

- 20 minutes lecture about today's experiment by the lecturer. See Syllabus last page and CANVAS under "files" for your assigned section. Only appear at your assigned section date and time. For example Section 3 & 4 both will assemble in Chemistry A room A224 at 8:00AM sharp (no late arrival) for short lecture on Thursday the second week to sixth week of classes. After 20 minutes of short lecture, Section 3 remains in the same Lab room A224 while Section 4 will assemble in adjacent Lab room A226 (in couple of minutes). Caution, check Canvas latest lab/class schedule.
- b. Immediately after assembly in **Room A224 or Room A226** (see Canvas and Syllabus for your assigned group & section), TA's will take attendance using attendance sheet and give 10-20 minutes orientation/instructions and a quiz on today's assigned experiment. Each group will give 3-5 minutes discussion session on their last week experiment experience and hands in the report for the previous week experiment. Each group can discuss the Abstract of their previous week experiment and pose couple of questions to other groups. One student of each group leads the discussion & alternated weekly among each group so each student will give at least one discussion session per experiment.
  - c. Each group will assemble by their assigned laboratory bench area and start conducting the experimental work for no later than 2 ½ hours. While at the start of the lab, the TA will examine the uncompleted and typed or handwritten **After-Lab Report (ALR)** that includes a completed **Pre-Lab Exercises (PLE)** section of this week experiment. Each student prepares ahead of class all parts of the ALR as instructed later in this syllabus that includes the PLE by reading and following the laboratory manual for the experiment (posted on Canvas) and answering questions prior to each laboratory to make sure that the student is well aware and familiar with the entire experiment. The background, introduction, & theory should be your own words as much as possible. Further report instructions is in the laboratory manual posted on Canvas.
  - d. While conducting the experimental work, each student & group must complete the recording of their experimental results on their lab report and on a TA's provided **datasheet** (sample datasheets are given in the manual at the end of each experiment). **Make sure you turn in the datasheet to the TA after you copy the data for yourself, before leaving the laboratory.** Following the completion of each laboratory, the students also complete the handwriting of their own **Post-Lab Questions (PLQ)**, based on lab manual instructions for the conducted experiment, to analyze their experimental results and groups communicate their findings during discussion. TA will check the data and have brief discussion before leaving the laboratory and may ask a student or a group to repeat suspicious or incorrect experimental results. **The PLE & PLQ must be turned in the following week as parts of the After-Lab Report (ALR) for each experiment or as announced by the Instructor/TA.**
  - e. The remaining half hour of the lab session and before dismissing the students, each student is responsible for general cleaning of their bench work area (including cleaning the glassware). Then 2 groups/week will alternate on a weekly basis to do more thorough cleaning (sink, floor, etc.) of the entire laboratory immediately before leaving the laboratory.

**Segment 3, Week 7 or 8:** Students assemble inside their chemistry lab or as instructed by Canvas announcement on their normal section schedule, to give **PPT presentation** on their work experience with one randomly select experiment (TA assigns randomly any of E1-E5), and **hand-in two reports**, last week **Experiment 5 report** and the **experimental design report** about their special project on chemical analysis of Ca content in commercial products.

**Segment 4, Tentative: Week 9, Final Exam:** Closed books & notes & no programmable calculator can be used. Check CANVAS for final exam schedule & location.

**Short Lectures on Experiments:** The short lectures (20 min.) occur prior to the start of a new topic and experiment. Refer to the course schedule (below) for specific dates and topics (refer to Segment 2 on first page of Syllabus).

**Lab Safety: Must follow safety instructions in lab manual (page ii, iii, and Appendix A) as posted on Canvas.**

**Office Hours Instructor / TA's :**

Lecturer	Room - Location	Office Hours Time	Contact
Dr. Ting Sun	Chemistry Bldg A Rooms A224 or A226	16:30-17:30 W and During W, TH lab sessions between 19:00-20:30	Ext. 4391 ting.sun@sjtu.edu.cn
TAs	See CANVAS or TA	See CANVAS or TA	

**Required Course Materials:** You must wear safety goggles through all your experimental work and whenever you are inside the laboratory and handling any chemicals. For calculation in laboratory, you will need a scientific calculator. However, programmable calculators are not allowed on the exams.

**Lab Manual (posted on CANVAS under “Resource”):** Based on edited version of Collaborative Investigations in Chemistry, Konigsberg Kerner & Penner-Hahn, Hayden McNeil; Hands on Chemistry Laboratory Manual, 1st Ed., Jeffrey A. Paradis, Kristen Spotz, McGraw Hill Higher Education Press, 2006.

**Course Methods:** You will conduct “inquiry” experiments where you are *not* expected to know the outcome in advance. A major goal of this lab course is to facilitate development of your qualitative reasoning skills. During this laboratory centered course you will be exposed to qualitative reasoning skills that scientists use when solving problems such as formulating hypothesis, organizing data, making inferences from data, and designing experiments. You will do some of your experiments individually and some in a group where you will combine and compare data, instead of competing with classmates for the “right” answer. In general, you will get no credit for memorizing “right” answers. Rather, your goal is to learn techniques for analyzing and interpreting data. A key objective is for you to learn *how* to approach a particular question (what data to measure, what chemicals should you use, etc.).

You don't need lab experience to do well in Chemistry Laboratory VC211. You *do* have to: prepare in advance for the labs; attend your laboratory section and work conscientiously and safely during the period; think about the experiments that you have done; and prepare in advance for the discussion. If each student do these things, then most of the students should be able to get at least a B grade for the course.

**In case of illness or other emergency:** Sometimes (rarely) students miss the exam because of illness or another emergency. If you are ill for the exam, e-mail your lecturer immediately and ask the Health Service or your M.D. for a note in confirmation.

**Absence and Make-ups:** Since the course is cumulative it is important that you do not miss a lab. Occasionally, circumstances will arise that force you to miss a lab. During spring term it may not be possible to schedule a make-up outside the normally scheduled lab hours. If you miss the last scheduled lab prior to check out, it will only be possible to schedule a make-up during the next year.

**Grading Policy (see Appendix C, Lab Manual on Canvas):** Your grade will be determined based on your performance in the laboratory, the final exam, class attendance and discussion. The weighted points are assigned as follows:

GRADING GUIDE	MAX. POINTS	MAX. % GRADE
<b>5 EXPERIMENTS 150 POINTS EACH AS FOLLOWING:</b> a. <b>PLE: PRE-LAB EXERCISES                   30 POINTS</b> <i>Including the pre-lab quiz grade.</i> b. <b>PLQ: POST-LAB &amp; DATA SHEET           40 POINTS</b> c. <b>EXPT'L OPERATION LAB WORK       50 POINTS</b> <i>“TA gives grade at end of experiment”</i> d. <b>ALR: AFTER-LAB REPORT               30 POINTS</b>	750	75%
<b>EXPERIMENTAL DESIGN REPORT: Relies on experiments 1 &amp; 3 analyzing calcium (Ca) in commercial products</b>	50	5%
<b>FINAL EXAM: CLOSED BOOKS &amp; NOTES</b>	150	15%
<b>FINAL PPT ON SELECT E1-E5 EXPERIMENT: Each group to present one assigned experiment during the 8<sup>th</sup> week of lab. You must document your experimental work with few photos to include in your reports and presentation.</b>	50	5%
<b>TOTAL</b>	<b>1000</b>	<b>100%</b>

**Honor Code Policy:** Experimental reports must be completed independently unless otherwise instructed. However students are encouraged to discuss their experimental experience but not copy from each other unless instructed by the TA to copy some of the experimental results. Exams must be worked independently and they are closed books & closed notes (not allowed: dictionaries, digital devices, computers, course materials, notes and textbooks, allowed: standard non-programmable calculator). Follow the instructions in the laboratory manual and as posted on Canvas. You are not permitted to download any document without the authorization of the instructor, however, you are allowed to hand write the information and instructions you need to complete your lab reports and assignments. You are not allowed to make copies of any course materials without the written permission of the original publisher. You must follow JI policy on honor code and review consequences for violation of such policy. Refer to your student handbook for further honor code policies.

**Lab Report Information and Turn in directions:** Detailed instructions to below sections are on CANVAS for VC211, under “Files/ Lab Manual & Experiments”. Students must underline and highlight titles & subtitles, i.e. **“Introduction”**, **“Theory”**, **“Procedure”**, **“Pre-Lab Exercises (PLE)”**, **Post-Lab Questions (PLQ)**, etc.

- A) Each student will be required to turn in for each experiment and **at the start of each experiment a printed & handwritten (combination as instructed below) individual comprehensive report** known as **After- Lab Report (ALR)**. The **ALR** will be initially incomplete because you still have to record your experimental data, notes and answering questions. The following **ALR** report sections should be completed as instructed below with only few clearly shown exceptions. TA inspects the **ALR** prior to the start of the experiment (see schedule in the next table):
1. **Typed Cover page** with experiment title, date, Group number, section number, your Chinese & Pinyin name, student ID, email address, and telephone number. Must also include under your name the names of your teammates in Chinese & Pinyin. If any of such information is missing then you may not be graded for the **ALR**.
  2. **Typed brief summary using your own words of the Introduction, Experimental Background, and Theory:** similar to the lab manual but use your own version as much as possible. All these sections of the report due upon entering the lab before conducting the same experiment. Give clear concise theory and procedure. Do not copy others ALR report or you may be in violation of honor code.
  3. **Pre-Lab Exercise (PLE): Typed or handwritten, ok.** Answer and complete all sections including providing data tables, as instructed in the lab manual for the PLE prior to entering the lab session. **TA will check the PLE and you cannot start any lab work if PLE is not completed.**
  4. **Post-Lab Questions (PLQ): Typed or handwritten, ok.** Must be completed and checked by the TA at the conclusion of each experiment before dismissal of the lab session. Ahead of lab session prepare all relevant sections including providing data tables, as instructed in the lab manual for the PLQ.
  5. **TA-s Datasheet:** Report all the experimental results on your report and on the provided TA’s datasheet. Have the TA check your data results before dismissal of the lab session. You must have correct record, reasonable result and keep tidy & safe. Gather all above report sections and attach to the next section.
  6. **Discussion of results, analysis, conclusions and recommendations:** These sections must be completed at the start of the next week experiment except for the last E5 experiment it will be due the same day you conduct the experiment. ALR should include data processing, analysis of results, discussion & correlation to theory, conclusions & recommendations (also complete exercise questions).
  7. **After-Lab Report (ALR):** All above sections are to be submitted to the TA as a final ALR report for each experiment. This final ALR report for each experiment is due the following week prior to the start of the next experiment except E5 will be due immediately after completing the experiment (the same day).
- B) Laboratory discussion, Final PPT on randomly selected experiment (TA assigns from E1-E5), and Final Report Experimental Design for Ca –analysis: See below table for requirements & due dates.

**VC211 SPRING 2019 LABORATORY COURSE SCHEDULE**  
**TENTATIVE SCHEDULE SUBJECT TO CHANGE**

**Class is divided into 12 sections, each section to report to the labs according to the following schedule:**

**S1/S2: Wed. 08:00-11:00**

**S3/S4: Wed. 18:00-21:00**

**S5/S6: Thur. 18:00-21:00**

**S7/S8: Frid. 08:00-11:00**

**S9/S10: Frid. 13:00-16:00**

**S11/S12: Frid. 18:00-21:00**

WK	Dates	Experiment Topics: Lab dates W, Th, F	Lab Manual Pages
1	Wed. Feb. 27	<b>Introduction and Safety Rules – one lecture, no labs</b> 14:00-15:40, at Dong Shangyuan building Room 100	<b>MANDATORY LECTURE</b>
2	Mar. 6-8	<b>E1: Acids and Bases</b> 1.Relative Acidity/Basicity of Common Household Products 2. Acid-Base Titration of vinegar.	<b>PLE1, PLQ1 DUE</b>
3	Mar. 13-15	<b>E2: Properties of Buffers</b> 1.Strong and Weak acids 2. Properties of a buffer 3.Designing a Buffer 4.Determination of Buffer Capacity	- <b>ALR1 E1 REPORT DUE</b> - <b>PLE2, PLQ2 DUE</b>
4	Mar 20-22	<b>E3: Spectrophotometric Analysis</b> 1.Adjusting the Spectrophotometer 2. Preparation of Standard Solutions 3.Making the Calibration curve Using the Standard Solutions 4. Determination of Unknown Concentration.	- <b>ALR2 E2 REPORT DUE</b> - <b>PLE3, PLQ3 DUE</b>
5	Mar 27-29	<b>E4(I)&amp;E4(II): Introduction to Kinetics, Determining the Rate Law</b> 1.Effect of Changing the Concentration of Reactants 2. Effect of Changing the Temperature 3.Effect of Adding a Catalyst 4. Designing Reactions to Determining the Reaction Order.	- <b>ALR3 E3 REPORT DUE</b> - <b>PLE4(A&amp;B), PLQ4(A&amp;B) DUE</b>
6	Apr. 3-5	<b>E5: Precipitation and Water Purity</b> 1. What is the Precipitate? 2. Precipitation Studies A. Is Precipitation Predictable? 3. Concentration and precipitation 4. Solvent Pollution and Precipitation. Individual reports due, each student must submit one group report immediately after conducting the experiment.	- <b>ALR4 E4(A&amp;B) REPORT DUE</b> - <b>PLE5, PLQ5 DUE</b> <b>ALR5 E5 TEAM REPORT DUE</b>
7	Apr. 10- 12	<b>NO LAB (no labs and no VC211 class meeting) / Prepare for next week your final report on Ca analysis and final presentation on select experiment.</b>	- <b>STUDY &amp; ASSIGNMENTS PREPARATION</b>
8	Apr 17-19	<b>Final PPT presentation</b> on select experiment & Final Report on Ca analysis in Ca-commercial products. <b>Students/Groups to appear either on their labs or as scheduled by your TA on Canvas.</b>	- <b>FINAL PPT DUE</b> - <b>FINAL REPORT EXPT. DESIGN: Ca- ANALYSIS DUE</b>
9	TBD	<b>Final Exam: Time &amp; location see CANVAS</b>	<b>Test schedule may change</b>

“For TA-s only (not students): lab orientation on each experiment for 5 weeks starting the second week at 2:00PM-5:00PM every Wed.”



# EXPERIMENT E1

## Chemistry of the Kitchen - Acids and Bases

Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department

(Modified version of D. Van Dinh and University of Michigan General Chemistry Laboratory Manual)

### I. OBJECTIVES

- Classify common household chemicals as either acids or bases using a homemade indicator solution.
- Develop an understanding of pH scale.
- Examine the difference between strong and weak acids.
- Titrate a sample of vinegar to determine the concentration of acid.

### II. INTRODUCTION

Take a moment and look around your house. Many every day chemicals used in your home are acids or bases. Some of most common acids and you may recognize and use includes vinegar (acetic acid), lemon juice (citric acid), and ammonia (some cleaners). Acids and are essential substances in home, industry and the environment. For example, the vast quantity of sulfuric acid manufactured in the United States each year is needed to produce fertilizers, polymers, steel, and many other materials. The influence of the acids on the environment can be seen through acid rain, which has caused numerous historic buildings and monuments to erode. If you have ever had a goldfish, you know how important it is to monitor and control the acidity of the water in aquarium (Figure 1). A characteristic that acids and bases share is their ability to turn certain organic compounds, such as vegetables materials, distinctive colors. Utilizing this knowledge, what are some methods in which you could test the acidity of the water in your goldfish aquarium?



Figure 1. A healthy fish tank requires careful control of acidity levels.

### III. BACKGROUND

#### A. Properties of Acids and Bases

Since the 17<sup>th</sup> century, acids and bases have been characterized by their sour and bitter taste, respectively. In modern chemistry, these concepts have taken on considerably more precise meaning. In fact, there are three definitions of acids and bases, the classical (Arrhenius), the Brønsted – Lowry, and the Lewis, which greatly expand our knowledge in these chemicals. Acids and bases differ greatly in their strength in water, that is, in the amount of  $\text{H}_3\text{O}^+$  or  $\text{OH}^-$  produced per mole of substance dissolved. They are generally as either strong or weak, depending on their dissociation into ions in water. Acids and bases are electrolytes in water, so the classification of acid and base strength correlates with the classification of the electrolyte strength: strong electrolyte such as strong acids dissociate completely (Figure 2), and weak electrolytes such as the weak acids undergo partial dissociation (Figure 3).

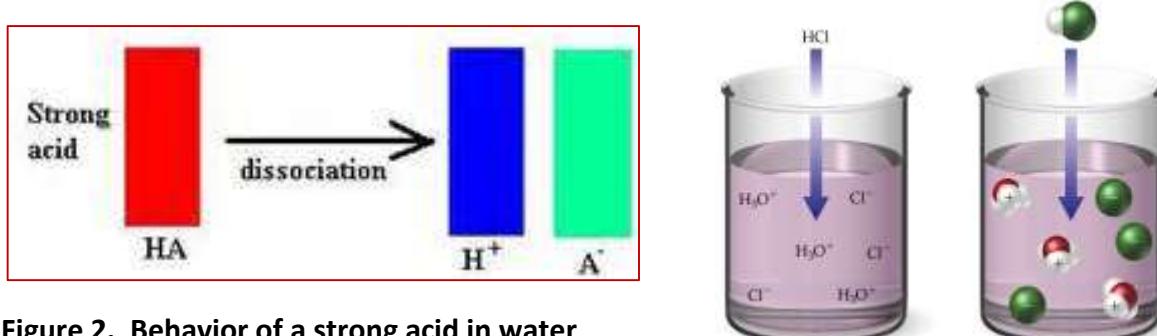
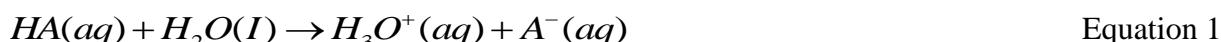


Figure 2. Behavior of a strong acid in water



In an aqueous solution of a strong acid, virtually no HA molecules are present (Equation 1).



At equilibrium, the  $[\text{H}_3\text{O}^+] = [\text{A}^-] = [\text{HA}]_{\text{initial}}$  and the  $[\text{HA}]_{\text{equilibrium}} = 0$ . Then the equilibrium constant for strong acid is essentially very large ( $K_a = [\text{H}_3\text{O}^+][\text{A}^-]/0$ , very large). Since this process essentially goes to completion, a single arrow is used ( $\rightarrow$ ) in the case of strong acids.

The situation is different in the case of a weak acid. From Figure 3, the majority of HA molecules in a solution of a weak acid are undissociated.

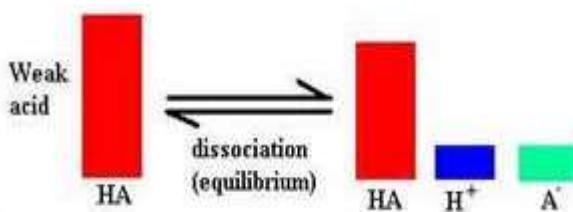
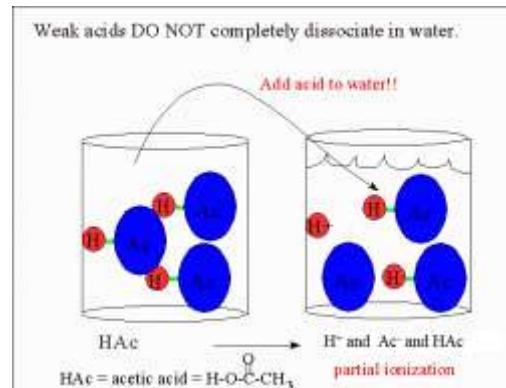
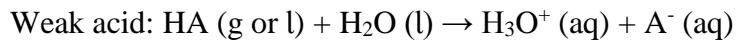
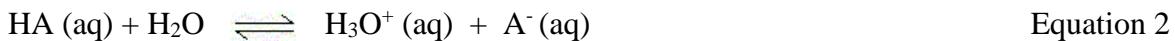


Figure 3. Behavior of a weak acid in water





Thus,  $\left[\text{H}_3\text{O}^+\right] \ll [\text{HA}]_{\text{initial}}$  and  $[\text{HA}]_{\text{equilibrium}} < [\text{HA}]_{\text{initial}}$  because the amount of acid dissociated is insignificant compared to initial concentration. Notice in Equation 2 below that the reaction is expressed as an equilibrium ( $\rightleftharpoons$ ) indicating that the reaction does not necessarily produce 100% products. In fact in the case of most weak acids, less than 5% of original HA molecules will actually dissociate to produce  $\text{H}_3\text{O}^+(\text{aq})$  and  $\text{A}^-(\text{aq})$  ions.



The percentage of acid that is actually dissociated can be quantified in terms of the acid dissociation constant,  $K_a$ , whose expression for the dissociation of a general weak acid, HA, in water is shown in Equation 3. Notice  $[\text{H}_2\text{O}]$  does not show up in the form of  $K_a$ .

At equilibrium:

$$K_a = \frac{\left[\text{H}_3\text{O}^+\right]\left[\text{A}^-\right]}{[\text{HA}]} \quad \text{Equation 3}$$

$K_a = x \cdot x / ([\text{HA}]_{\text{init}} - x)$  = small. Since  $x$  is insignificant compared to  $[\text{HA}]_{\text{init}}$ , then  $K_a = x^2 / [\text{HA}]_{\text{init}}$ .

Like any equilibrium constant, the magnitude of  $K_a$  tells how far to the right the reaction has proceeded when equilibrium is reached. Thus, the stronger the acid, the higher the  $[\text{H}_3\text{O}^+]$  at equilibrium, the larger the  $K_a$ :

Stronger acid  $\Rightarrow$  higher % HA dissociation  $\Rightarrow$  higher  $[\text{H}_3\text{O}^+]$   $\Rightarrow$  larger  $K_a$

Likewise, the weaker the acid, the smaller the  $K_a$ :

Weaker acid  $\Rightarrow$  lower % HA dissociation  $\Rightarrow$  lower  $[\text{H}_3\text{O}^+]$   $\Rightarrow$  smaller  $K_a$

## B. The pH Scale

In aqueous solutions,  $[\text{H}_3\text{O}^+]$  can vary over an enormous range: from about 10 M to  $10^{-15}$  M. To handle numbers with negative exponents more conveniently in calculations, we convert them to positive numbers using a numerical system called p-scale, the negative of the common (base 10) logarithm of the number. Applying this numerical system to  $[\text{H}_3\text{O}^+]$  gives pH, the negative logarithm of  $[\text{H}_3\text{O}^+]$  or  $[\text{H}^+]$  (Equation 4).

$$\text{pH} = -\log[\text{H}_3\text{O}^+] \quad \text{Equation 4}$$

For example, a solution with a  $[H_3O^+]$  of  $5.4 \times 10^{-4} M$  has a pH of 3.27 (Equation 4a).

$$pH = -\log[H_3O^+] = -\log(5.4 \times 10^{-4}) = 3.27 \quad \text{Equation 4a}$$

Likewise, the pH of a  $1.0 \times 10^{-12} M H_3O^+$  solution is 12.00 on the pH scale. Note that the higher the pH, the lower the  $[H_3O^+]$ . Therefore, an acidic solution has a lower pH (higher  $[H_3O^+]$ ) than a basic solution (see for example in Figure 4 how abundance ratio of  $\text{CH}_3\text{COOH}$  drops with pH while the  $\text{CH}_3\text{COO}^-$  increases with pH). At  $25^\circ C$  the  $[H_3O^+]$  in pure water is  $1.0 \times 10^{-7} M$ , so the pH of pure water at  $25^\circ C$  is 7.00 and aqueous solutions typically fall within range 0 to 14. This information is summarized in Table 1.

Type of solution	pH
Basic	$> 7.00$
Neutral	$= 7.00$
Acidic	$< 7.00$

Table 1. pH of acids & bases

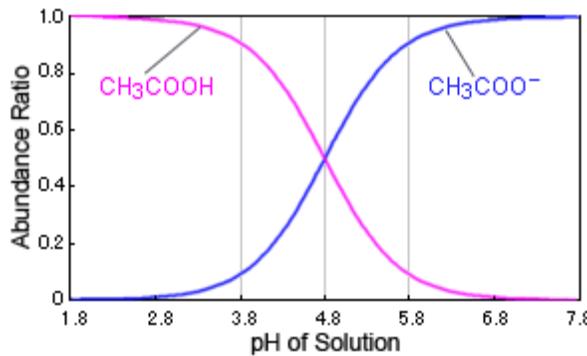


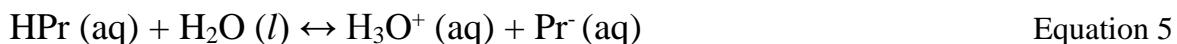
Figure 4. Acetic acid abundance ratio with pH

In the laboratory, pH values are usually obtained with an acid-base indicator or with a more precise instrument called a pH meter.

### C. Calculating the pH

It is possible using what we have learned so far to calculate the theoretical pH of any aqueous acidic solution without using a pH meter. In the case of a strong acid, our job is simple since we can assume that the concentration of  $[H_3O^+]$  is the same as the starting concentration of the strong acid and we simply use the calculation from Equation 4. However for weak acids the amount of acid partially dissociated to  $[H_3O^+]$  is much smaller than the amount of acid itself and can be calculated just from knowing the  $K_a$  and the initial concentrations of the weak acid. Giving the  $K_a$  and  $[HA]_{initial}$  of the weak acid to calculate  $[H_3O^+]$ , we can calculate the pH. As an example, we will calculate the pH of 0.10 M solution of propanoic acid ( $\text{C}_2\text{H}_5\text{COOH}$ , which we symbolize as HPr). A good first step in doing these problems is to look up the value of the  $K_a$ . In the case of propanoic acid,  $K_a = 1.3 \times 10^{-5}$  at  $25^\circ C$ .

In order to determine the  $[H_3O^+]$  of this solution, we must next write the balanced equation and  $K_a$  expression, as in Equation 2 (Equation 5) and 3 (Equation 5a), respectively.



$$K_a = 1.3 \times 10^{-5} = \frac{[H_3O^+][Pr^-]}{[HPr]} \quad \text{Equation 5a}$$

From Equation 5, we see for each mole of HPr that dissociate one mole of  $H_3O^+$  and  $Pr^-$  each produced. Knowing this and given  $[HPr]_{initial}$  (0.10 M) we can set up a reaction table (Table 2), commonly called an I.C.E. table (Initial Change Equilibrium table).

	HPr (aq)	+ H <sub>2</sub> O (l)	$\leftrightarrow$	$H_3O^+(aq)$	+ Pr <sup>-</sup> (aq)
Initial	0.10 M	-		0	0
Change	- $x$	-		+ $x$	+ $x$
Equilibrium	0.10M - $x$	-		$x$	$x$

**Table 2. Weak acid concentration calculations at equilibrium**

In the “Initial” row we include the starting concentration of the weak acid. Note that the concentration of  $H_3O^+$  and the conjugate base  $Pr^-$  are both initially equal to zero. Because we don’t yet know how much of the HPr dissociates, we express the changes in terms of variable,  $x$ . we know that how much HPr is lost ( $-x$ ) in the “Change” row, we must gain the same amount: ( $+x$ ) of  $H_3O^+$  and  $Pr^-$ . The “Equilibrium” row is the sum of the “initial” and “Change” rows.

We are now ready to substitute information from our “Equilibrium” row into the  $K_a$  expression and solve for  $x$  (Equation 5b).

$$K_a = \frac{[H_3O^+][Pr^-]}{[HPr]} = 1.3 \times 10^{-5} = \frac{(x)(x)}{(0.10-x)} \quad \text{Equation 5b}$$

Since  $K_a$  is small for HPr, it dissociates very little so the value of  $x$  is negligibly small compared to 0.10 M; therefore, we avoid solving the quadratic equation by approximating  $0.10 - x = 0.10$  M (Equation 5c)

$$1.3 \times 10^{-5} = \frac{(x)(x)}{(0.10)} \quad \text{Equation 5c}$$

$$x = \sqrt{(0.10)(1.3 \times 10^{-5})} = 1.1 \times 10^{-3} M = [H_3O^+] \quad \text{Equation 5d}$$

Equation 6 shows that now that we have found the  $[H_3O^+]$  in our solution of 0.10 M propanoic acid, we can calculate its pH using Equation 4.

$$pH = -\log[H_3O^+] = -\log(1.1 \times 10^{-3}) = 2.96$$

Equation 6

Handling log significant figures (SF):  $\log 1.1$  (2SF all) = 2.96 (2SF beyond decimal point). Anti  $\log 2.96$  (2SF beyond decimal point) = 1.1 (2SF all).

This mathematical process can also be reversed. For example, an environmental chemist can measure the experimental pH and use it to determine the concentration of acid in a sample of acid rain.

#### D. Titration

In addition to the method described above, the concentration of an acid can also be determined experimentally by an acid – base titration. In an acid – base titration, a measured volume of acidic solution of unknown is placed in a flask beneath a burette containing the known (standardized) base solution (Figure 5). A few drops of acid – base indicator, usually phenolphthalein, are added to the flask containing the acidic solution. The standardized solution of base is then added slowly to the flask until the end point is reached. The end point of the titration occurs when the indicator changes color permanently due to the presence of excess  $OH^-$  ions (phenolphthalein is colorless in acid and pink in base). Knowing the stoichiometry of the acid – base reaction and the amount of base used to reach the end point, scientists can discover the unknown  $[H_3O^+]$  of the acidic solution.



Figure 5. Experimental setup for an acid-base titration

#### E. Overview

In this series of experiments, you will examine several of various methods in which the pH of a solution can be measured. Knowing that acids and bases possess the ability to change the color of certain indicator solutions, you will use red cabbage extract and a universal indicator in Part A as acid – base indicators. You will develop a pH scale based on the fact that red cabbage extract changes colors when mixed with different household chemicals. In Part B, you will study the differences between strong and weak acids using pH meter. Using this knowledge, you will then determine an unknown concentration of a weak acid. In Part C, you will calculate the concentration of acid in vinegar by performing an acid – base titration.

**IV. EXPERIMENTAL PROCEDURES: “Make sure you take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5”**

**Part A. Relative Acidity/ Basicity of Common Household Products**

Chemicals used	Materials used
<b>Universal indicator papers:</b> Strips of universal pH indicator paper. <b>Various household chemicals:</b> (example ammonia, vinegar, Shampoo (colorless), soda (a colorless variety), milk, lemon juice, liquid detergent (without dyes), milk of magnesia, tap H <sub>2</sub> O, deionized H <sub>2</sub> O, Bleach)	Several beakers of various sizes 10-mL Graduated cylinder Glass stirring rod Test tube w/rubber stopper Test tube rack Funnel Hot plate & Knife

1. Working alone, select one or two household products to test from the available samples. Rinse a freshly washed beaker with 5 mL of your selected household chemical(s).
2. Pour 10 mL of the same household product into the beaker(s). Immerse partially a strip of universal pH indicator paper, then let the indicator paper(s) dry and label it with the sample that you tested. Repeat step 1 once again.
3. Label your samples and bring the indicator paper to the front of the lab for observation by the class. Be sure to record results for other household products (color of indicator paper and corresponding pH from the indicator paper box or from the chart posted on laboratory benches).
4. Repeat procedures (2.&3.) to test the pH of a stock solution of 1M NH<sub>3</sub>.H<sub>2</sub>O (inside eye-dropper vial) but use directly only 1-2 drops.

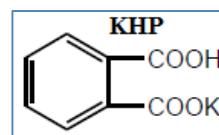
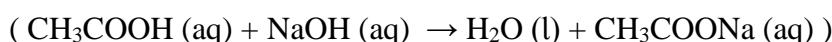
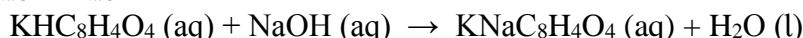
**For the remaining procedures parts B & C, students work in groups as assigned by instructor where each two students in a group works together.**

**Part B. Concentration of unknown molarity of NaOH solution using KHP titration**

**Introduction**

Potassium hydrogen phthalate (KHP, KHC<sub>8</sub>H<sub>4</sub>O<sub>4</sub>) can be used to titrate the concentration of NaOH solution. (Acetic Acid (HAc): CH<sub>3</sub>COOH)

$$C_{\text{NaOH}} V_{\text{NaOH}} = m_{\text{KHP}} / M_{\text{KHP}}$$



Chemicals used	Materials used
Unknown molarity NaOH solution inside bottle labelled as 0.1M NaOH. KHP pH indicator (phenolphthalein)	Provided glassware: beakers, Erlenmeyer flasks, & graduation cylinders Weighing papers & desiccators

## Procedure for Part B

1. At a designated area, wear protective gloves and wash the provided glassware of your group using detergent, brush and tap water and then rinse with de-ionized water, 3 times each. Be careful not to break or crack the glassware and not to injure yourself. Any broken or cracked glassware must be reported to the instructor and the glassware must be disposed carefully in the provided glass disposal box and not into the trash can or sink.
2. Using the Mettler balance, carefully weigh out 0.4000-0.6000g KHP into 3 labeled freshly washed Erlenmeyer flasks, record each weight. Add no more than 30-40 mL of de-ionized water to entirely solve KHP and 2-3 drops of phenolphthalein (to one of the flasks).
3. Run about 25.0 mL of deionized water through a 50-mL burette and then rinse the burette with about 5.0 mL of the labelled 0.1 M NaOH solution. Mount the burette in the burette stand.
4. Use a funnel to add roughly 30.0 mL of the labelled 0.10 M NaOH solution into the burette. Drain any air bubbles from the tip of the burette into a waste beaker. Record the initial volume  $V_0$  of the burette ( $-/+0.02\text{mL}$ ).
5. Slowly add the NaOH solution from the burette drop-wise to the KHP solution in the flask with the indicator, swirling the flask after each addition. Continue until the endpoint is reached. Record the final volume  $V_1$ .
6. Repeat steps 4-5 for a second and third trial using the other flasks with KHP solution.

## Part C. Acid – Base Titration of Vinegar Solution

$$\text{Calculation: } 10 C_{\text{NaOH}} V_{\text{NaOH}} = C_{\text{HAc}} V_{\text{HAc}}$$

Chemicals Used	Materials Used
Same solution bottle of NaOH of Part B. Vinegar with unknown concentration of acetic acid ( $\text{CH}_3\text{COOH}$ ) Phenolphthalein	Burette and burette stand 250-mL Erlenmeyer flask 100-mL Beaker (2) 25-mL Pipet w/bulb & graduated cylinder

1. Run about 25.0 mL of deionized water through a 50-mL burette and then rinse the burette with about 5.0 mL of the labelled 0.1 M NaOH solution. Mount the burette in the burette stand. (no need to rinse if you are using the same burette and stock solution bottle of NaOH from Part B)
2. Use a funnel to add the labelled 0.1 M NaOH solution (from Part B) into the burette and fill it up to near below the top mark. Drain any air bubbles from the tip of the burette into a waste beaker. Record the initial volume  $V_1$  ( $\pm 0.02 \text{ mL}$ ).
3. Pipet 25.00mL of the vinegar into a 250.00mL volumetric flask, add de-ionized water to the calibration line and shake upside down several times.
4. Pipet 25.00 mL of the solution in step 3, into 3 freshly washed & rinsed Erlenmeyer flasks and add 2 – 3 drops of phenolphthalein to the flask.
5. Slowly add the NaOH solution, drop wise from the burette to the vinegar, swirling the flask after each addition. Continue until the endpoint is reached. Record the final volume  $V_2$  ( $\pm 0.02 \text{ mL}$ ).
6. Repeat steps 2 – 4 for a second trial and third trial.

#### **Part D. Closing Your Lab Session**

1. Wear protective gloves and wash the used glassware using detergent, brush and tap water and then rinse with de-ionized water. Be careful not to break or crack the glassware. Any broken or cracked glassware must only be disposed carefully into the provided glass disposal box
2. Clean up your designated working area thoroughly so the next lab session can use safely.
3. A designated team from the groups will be instructed just before dismissal of the laboratory to inspect and insure the general cleaning and safety of the laboratory.

**From now and on, you must follow Part D thoroughly when conducting any experimental work inside the lab area.**

## E1: Chemistry of the Kitchen: Acids and Bases

Name:	Lab instructor:
Date:	Lab Section:

### V. PRE-LABORATORY EXERCISES (PLE)

1. Define the underlined words in the **BACKGROUND** section.
2. Determine the concentration of  $H_3O^+$  in a solution with a pH of 9.78.
3. Given solutions of the same concentration, which would you expect to have a lower pH, a strong or weak acid? Explain.
4. In this laboratory experiment you will be calculating the concentration of an unknown sample of acetic acid using the pH of the sample and the  $K_a$  for acetic acid. Use your textbook to find the  $K_a$  for acetic acid. Be sure to record this value in your notebook so you will have it available during the experiment.
5. You will be performing a titration of vinegar (an aqueous solution of acetic acid,  $HC_2H_3O_2$ , also more commonly known as  $CH_3COOH$ ) in this laboratory experiment. To prepare you for this titration, please read the section on acid – base titrations in your textbook and then do the following:

- a) Write the balanced molecular equation and the net ionic equation for the neutralization reaction between aqueous acetic acid and aqueous sodium hydroxide.
- b) You place 10.00 mL of a  $\text{HC}_2\text{H}_3\text{O}_2$  solution of unknown concentration in a flask and add a few drops of indicator. You then titrate the acid with 0.24 M NaOH. If the initial reading on the burette was 0.19 mL and the final reading was 26.50 mL, what is the concentration of the  $\text{HC}_2\text{H}_3\text{O}_2$  solution?
- c) Some solutions (such as vinegar) are commonly reported in terms of percent by mass. Assuming the density of the acetic acid solution you found in question 6b is the same as the density of pure water at  $25^\circ\text{C}$ , determine the percent by mass of the vinegar in the sample.
6. Using your notes from CHM110 and literature, write the equation that predicts the effect of temperature on equilibrium constant, then estimate to the equilibrium constant of acetic acid at  $12^\circ\text{C}$  by using its known value at  $25^\circ\text{C}$ .

## E1: Chemistry of the kitchen: Acids and Bases

Name:	Lab instructor:
Date:	Lab section:

## VI. RESULTS AND POST – LABORATORY QUESTIONS (PLQ)

### Part A. Relative Acidity/Basicity of Common Household Products

Attach a table summarizing the colors of the universal indicator paper that you used to test the pH of the selected one or two household products. Put a star next to the household indicator product(s) that you personally tested. Your table should include a column that correlates the color of the universal indicator paper with the pH of the solution (this information is found on the box of the universal indicator paper or on a chart placed at the work benches).

### Part B. Concentration of Unknown Molarity of NaOH Solution

Trial	1	2	3
$m_{KHP}$ (g)			
Total Vol. of NaOH (mL)			
$C_{NaOH}$ (mol/L) = $m_{KHP} \times 1000 / (MW_{KHP} \times V_{NaOH})$			
Average $C_{NaOH}$ (mol/L)			

\*Show your work for the calculation of the concentration of the unknown NaOH and compare to the value on the label of the used NaOH bottle.

### Part C. Acid – Base Titration of Vinegar: MW<sub>KHP</sub> = 204 g/mol

	Initial Vol. of NaOH (mL)	Final Vol. of NaOH (mL)	Total Vol. of NaOH (mL)
Trial 1			
Trial 2			
Trial 3			
Average			

Show your work for the calculation of the molarity of acetic acid vinegar.

Show your work for the calculation of the % by mass of acetic acid in vinegar. How does this value compare to the value on the bottle of vinegar?

**SAMPLE DATASHEET FOR A LAB SECTION**  
**(FOR REFERENCE ONLY, STUDENTS TO OMIT THIS PAGE)**

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P														
1																														
2	<b>VC211 DATASHEET FOR EXPERIMENT: E1 ACIDS &amp; BASES</b>												1 Dilution/2STDNTS & then titrate 25.00mL a flask																	
3	SECTION: _____			TA: _____			LAB ROOM: _____																							
4																														
5																														
6				Household pH			KHP Accurate Weight			KHP Titration			Acetic Acid Titration																	
7				2 pH/2STDNTS			3 FLASKS / 2 STDNTS			3 FLASKS / 2 STDNTS			3 FLASKS / 2 STDNTS																	
8	GRP	NAME	ID	HSHLD	NH <sub>3</sub> .H <sub>2</sub> O	KHP	KHP	KHP	NaOH	NaOH	NaOH	NaOH	NaOH	NaOH	NaOH															
9	#	Chinese		pH	pH	(g)	(g)	(g)	V1 (mL)	V2 (mL)	V3 (mL)	V4 (mL)	V5 (mL)	V6 (mL)																
10																														
11																														
12																														
13																														
14																														
15																														
16																														
17																														
18																														
19																														
20																														
21																														
22																														
23																														
24																														
25																														
26																														
27																														

## EXPERIMENT E2

### The Properties of Buffers: Resisting Change in a Turbulent World

Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department

(Modified version of D. Van Dinh and University of Michigan General Chemistry Laboratory

Manual) [NEXT YEAR MODIF TO DESIGN & TEST 2 BUFFERS INSTEAD OF 4 &  
MODIFY PROCEDURE TO INCLUDE pH# CORRESPONDING TO THE PLQ  
DATASHEET]

#### I. OBJECTIVES

- Investigate strengths of acids
- Learn how to use pH meter
- Investigate how buffers work.
- Prepare a buffer solution with a target concentration and pH.
- Determine the buffer capacity of a solution.



#### II. INTRODUCTION

Many different systems require the control of the conditions so that radical changes do not occur when the system is stressed in some way. For example, our body temperatures remain stable in hot and cold days through perspiration and increased metabolism. In doing so, the effect of stress due to temperature changes is lessened. In everyday language, a buffer is something that lessens the impact of an external force. One of the most important examples of a system requiring controlled conditions through a buffer is our blood which is a  $\text{H}_2\text{CO}_3/\text{HCO}_3^-$  system. Human blood requires a pH between 7.35 and 7.45. If the pH of the blood drops below 6.9 or rises above 7.8, then death is likely. Even within the “safe” range, the optimum pH for many bodily processes is quite narrow; therefore slight disturbances to the pH can significantly impair normal processes. A disturbance that can severely alter the blood pH is cardiac arrest. When the heart stops, metabolic acidosis sets in. Lactic acid and  $\text{CO}_2$  cannot be removed from the blood and the pH is dramatically lowered. By applying their knowledge of buffers, hospital emergency room personnel are able to quickly administer the fluids necessary to help restore the victim’s blood to its normal state and stave off death (Figure 1). How do they know what concentration of fluids to use in order to restore the blood pH to its normal range?

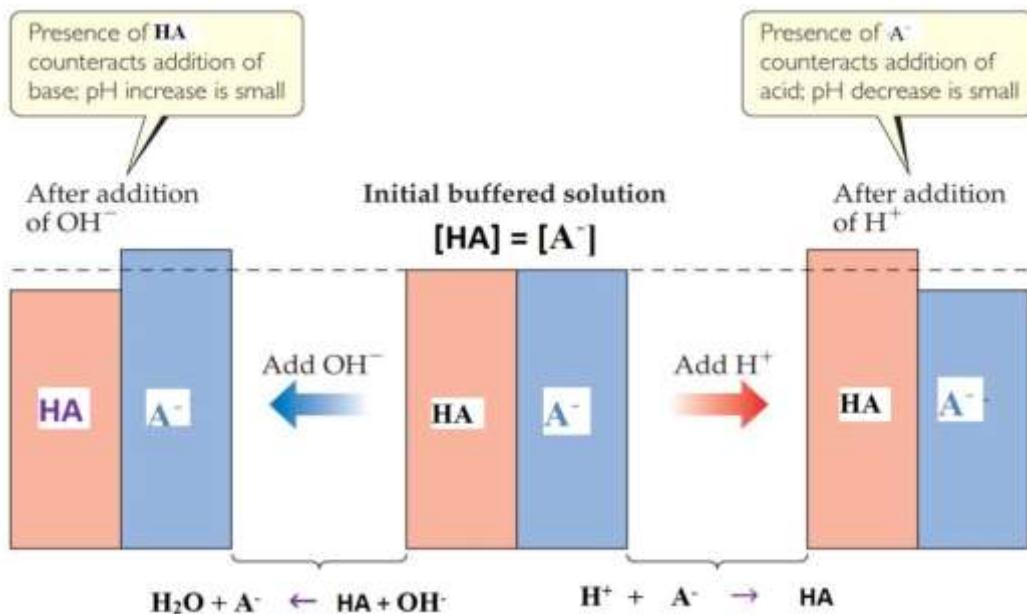
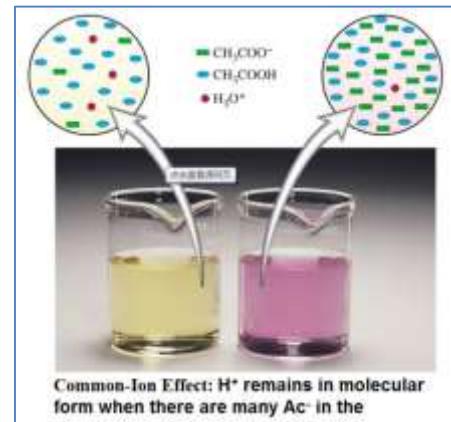


**Figure 1. A buffered intravenous drip**

### III. BACKGROUND

#### A. The properties of Buffers

Chemists apply the concepts of a buffer to solutions with the ability to resist changes in pH. In many areas of research, chemists need an aqueous solution that resists a pH change when hydrogen ions from a strong acid, or hydroxide ions from a strong base, are added. An acid – base buffer is able to resist changes in its pH by containing an acidic component that can neutralize added  $\text{OH}^-$  ions and a basic component that can neutralize added  $\text{H}^+$  (or  $\text{H}_3\text{O}^+$ ) ions. Most commonly, the components of a buffer are the conjugate acid – base pair of a weak acid. For example, an aqueous mixture of weak acid such as acetic acid,  $\text{CH}_3\text{COOH}$ , and its conjugate base acetate,  $\text{CH}_3\text{COO}^-$ , (simply formed by a solution of sodium acetate added to the weak acid). Buffers work through a phenomenon known as the common – ion effect. The essential feature of a buffer is that it consists of high concentrations of the acidic ( $\text{CH}_3\text{COOH}$ ) and basic  $\text{CH}_3\text{COO}^-$ . This allows the relative concentrations of the buffer components to stay about the same when small amounts of  $\text{H}^+$  or  $\text{OH}^-$  ions are added to the buffer: the acidic component will neutralize any added base and the basic component will neutralize any added acid (Figure 2).





**Figure 1. A buffer works by neutralizing small amounts of added acid or base**

It is important to note that a buffer will only work when the amount of  $\text{H}^+$  and  $\text{OH}^-$  added is much smaller than the amounts of acid – base components of the buffer present. The amount of strong acid or strong base that can be added to a buffer depends on its buffer capacity. The added ions have little effect on the pH because one or the other buffer components consumes them. To help you better understand this concept, let's look at an example.

Consider what happens when small amounts of strong acid or base are added to a buffer containing high  $[\text{C}_2\text{H}_5\text{COOH}]$  and  $[\text{C}_2\text{H}_5\text{COO}^-]$ . As a weak acid, propanoic acid dissociates only slightly in water (Equation 1):



The expression for the acid dissociation constants is:

$$K_a = \frac{[\text{H}^+] [\text{C}_2\text{H}_5\text{COO}^-]}{[\text{C}_2\text{H}_5\text{COOH}]}$$

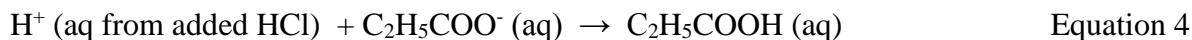
Equation 2

Noting that  $K_a$  is a constant and solving for  $[\text{H}^+]$ , we see that the  $[\text{H}^+]$  of a solution is dependent on the conjugate acid – base pair concentration ratio:

$$[\text{H}^+] = K_a \times \frac{[\text{C}_2\text{H}_5\text{COOH}]}{[\text{C}_2\text{H}_5\text{COO}^-]}$$

Equation 3

From Equation 3, you can see that if the ratio of the acid to base increases, the  $[H^+]$  increases. Likewise, if the ratio of the acid to base decreases, the  $[H^+]$  decreases. When a small amount of strong acid is added, such as HCl, the increased amount of  $H^+$  reacts with a nearly stoichiometric amount of  $C_2H_5COO^-$  in the buffer to form more  $C_2H_5COOH$ . (Equation 4)



As a result, the  $[C_2H_5COO^-]$  goes down by the amount of  $[H^+]$  and the  $[C_2H_5COOH]$  goes up by the same amount. This increases the concentration ratio of the acid to base components of the buffer by a slight amount, resulting in a small pH drop. Adding a small amount of a strong base, such as NaOH, produces the opposite results (pH decreases with increased acidity or increases with increased basicity,  $pH = -\log [H^+]$ ).

### B. Preparing a Buffer

As stated in the introduction, many times emergency room personnel are required to administer a buffer solution that simulates the exact concentration and pH of normal human blood. How do they prepare a buffer with a specific concentration and particular pH? In order to calculate the expected pH for a buffer, the **Henderson–Hasselbalch** equation is used. The Henderson–Hasselbalch equation is a mathematical expression useful for buffer calculations. Taking the negative logarithm of both sides of Equation 3 gives:

$$-\log[H^+] = -\log K_a - \log \frac{[C_2H_5COOH]}{[C_2H_5COO^-]} \quad \text{Equation 5}$$

Substituting definitions of  $pH$  and  $pK_a$  into Equation 5, we obtain:

$$pH = pK_a + \log \frac{[C_2H_5COO^-]}{[C_2H_5COOH]} \quad \text{Equation 6}$$

In general, the Henderson–Hasselbalch equation for any conjugate acid–base pair can be written as:

$$pH = pK_a + \log \frac{[\text{base}]}{[\text{acid}]} \quad \text{Equation 7}$$

In preparing a buffer, the correct  $[\text{base}]/[\text{acid}]$  ratio must be determined. Knowing the target pH, one can use the Henderson–Hasselbalch equation in order to calculate the correct concentrations of acid–base ratio to use in a buffer. The ratio gives some idea of the stability of the buffer. The most stable buffers have a ratio of 1:1. This ratio and the target pH are vital to know because it helps us to determine the conjugate acid–base pair to use when preparing a buffer.

For example, imagine you have to prepare 0.250 L of a pH = 5.19 buffer solution of propanoic acid with a total concentration of 0.0500 M from stock solutions of 0.100 M propanoic acid and 0.100 M sodium propanate ( $C_2H_5COONa$ ). Propanoic acid ( $C_2H_5COOH$ ) has a  $pK_a$  of 4.89.

To determine the correct  $[C_2H_5COO^-]/[C_2H_5COOH]$  ratio, rearrange Equation 6 to obtain:

$$pH - pK_a = \log \frac{[C_2H_5COO^-]}{[C_2H_5COOH]} \quad \text{Equation 8}$$

$$5.19 - 4.89 = 0.30 = \log \frac{[C_2H_5COO^-]}{[C_2H_5COOH]} \quad \text{Equation 8a}$$

$$10^{0.30} = 2.00 = \frac{[C_2H_5COO^-]}{[C_2H_5COOH]} \quad \text{Or} \quad \text{Equation 8b}$$

$$[C_2H_5COO^-] = 2.00[C_2H_5COOH] \quad \text{Equation 8c}$$

This tells us that the amount of  $[C_2H_5COO^-]$  must be twice the amount of  $[C_2H_5COOH]$ . Next, we determine the actual concentrations of the weak acid and the weak base to use in the buffer to meet the required concentration. From Equation 9, we can see that we must have the two buffer component concentrations equal to the total buffer strength.

$$[\text{buffer}] = [\text{acid}] + [\text{base}] \quad \text{Equation 9}$$

Using our example:

$$0.0500M = [C_2H_5COOH] + 2.00[C_2H_5COOH] \quad \text{Equation 9a}$$

$$0.0500M = 3.00[C_2H_5COOH] \quad \text{Equation 9b}$$

$$0.0167M = [C_2H_5COOH] \quad \text{Equation 9c}$$

$$[C_2H_5COO^-] = 2.00[C_2H_5COOH] = 0.0333M \quad \text{Equation 9d}$$

The final step in preparing the buffer is to mix the correct volume amounts of the conjugate acid–base pair to give the desired concentration. Knowing the concentrations calculated from Equations 9c-d, the final volume of solution needed (0.250 L) and the initial concentration of the stock solutions (0.100 M) we can use the formula,  $M_1V_1 = M_2V_2$ , to calculate the exact volume of stock solutions needed.



To find the volume of C<sub>2</sub>H<sub>5</sub>COOH to use in our buffer we calculate:

$$V_1 = \frac{M_2 V_2}{M_1}$$
 Equation 10

$$V_1 = \frac{(0.0167M)(0.250L)}{0.100M}$$
 Equation 10a

$$V_1 = 0.0418L$$
 Equation 10b

Repeating these steps to calculate the volume of C<sub>2</sub>H<sub>5</sub>COO<sup>-</sup> to use would give us 0.0833 L. Thus, we mix 41.8 mL of 0.100 M propanoic acid solution and 83.3 mL of 0.100 M sodium propanate solution in a 250.0- mL volumetric flask. Adding de-ionized H<sub>2</sub>O while mixing will give a total solution with a volume of 250.0- mL, a pH of 5.19 and a total concentration of 0.0500 M.

### C. Overview

In this lab you will learn how acid strength affects reactions with metals and be able to measure pH using pH meter. Also you will learn how to prepare a buffer of a desired pH and concentration. You will observe how buffers stabilize the pH of a solution even after adding a strong acid or strong base. Also, record the average temperature displayed by the pH meter for the entire experiment that you may use in your report discussion.

Safety is part of science, please protect your safety and the safety of others.

#### SAFETY WARNING:

Safety rules & chemical waste disposal guidelines must be followed in order to prevent personal injury, protect yourself, others & the environment. If you are unable to observe the rules then you are at risk of being dismissed from the lab.

#### CAUTION:

Do not dump any of the reagents down the sink! Discard the waste in an appropriate waste container under the supervision of your instructor! Reduce waste by working diligently and do not repeat experimental trials without the approval of the instructor. Do not allow solutions to come in contact with your skin! Wear gloves & goggles.



In Part A you will learn the strengths of acid using pH digital meter and also magnesium strips.

In Part B, you will be acquainted with the procedures and calculations involved in designing and preparing a buffer. Each student will learn to design acetic acid/sodium acetate buffer with varying pH. Using the knowledge gained from the calculations, you will then prepare your own buffer, given a total buffer concentration, a target pH, and known concentrations of conjugate acid–base pairs. With a pH meter you will measure the initial pH of each buffer.



Measuring pH using a digital pH meter.

Then in Part C, you will learn and test how the pH properties of each buffer changes with a small addition of strong acid and strong base and exchange data with other groups.

In Part D, you will titrate the buffer you made in Part B with a strong acid in order to determine its buffer capacity.

## **IV. EXPERIMENTAL PROCEDURES “confirm with the datasheet on the last page”**

- Make sure you take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5.
- Always freshly wash & rinse glassware with deionized water before use.
- If 50-mL beakers are not available then add enough solutions to each beaker to make sure the pH meter electrode tip is completely immersed in solution.
- Each pipet must be rinsed with deionized water, use rubber bulb pump to force excess water out, then wipe with clean tissue. Do not use any stock solution to rinse the pipet since this may cause waste of chemicals.

### **PART A: pH Meter Calibration (each group must do)**

- Accuracy of pH measurements depends on various factors including pH meter type, temperature, experimental procedure, concentration of chemicals, etc.
  - Make sure to conduct the entire experiment at minimum variation of room temperature hence the equilibrium constant and  $[H^+]$  depends on T (see Chapter 19 of VC211 course:  $\Delta G^0 = -RT\ln K$  where K &  $H^+$  are related in aqueous solution).
  - Minimize contamination of the pH meter electrode and when experiment is completed ensure the proper rinsing and storage of the electrode.
  - If pH meter electrode is not contaminated or misused then no need to recalibrate throughout the entire experiment of E2.
  - For further detailed instructions review the power point presentation for E2.
1. Using a clean beaker (50-mL) pour enough volume of the calibration stock buffer of pH = 4.003 (or as provided) to be able later to completely immerse the head of the pH meter electrode. The stock buffer pH may be different than 4.003 and also the stock buffer may be not the same buffer as that inside the storage bottle (where the electrode is normally stored).
  2. Turn on the pH meter, rinse electrode thoroughly with deionized water and carefully wipe the tip with clean dry tissue.
  3. Insert the electrode tip thoroughly into the 50-mL beaker buffer. Push the calibration button and wait (about 15 seconds) until the pH reaches 4.00 (in case another stock buffer is used then wait until the reading is very close to the stock solution pH).
  4. Carefully remove the electrode, rinse with deionized water, and wipe with clean dry tissue.
  5. Immediately stow away the electrode in its storage buffer bottle. Now the meter is ready to measure pH of all the samples for the entire experiment.

**Warning: If you intentionally or ignorantly do not rinse the electrode with deionized water and wipe the tip, then you may contaminate the electrode and cause erroneous errors in pH measurement for the remaining samples. Under no circumstance the pH meter is left to dry out of its proper storage buffer bottle. In addition you may be subject to heavy grading penalty and be liable for the intentional damage of the meter. So please read procedures very carefully and follow the lab instructions.**

## PART A.1: pH of Strong and Weak Acids

- Modified version of the original procedure from Univ. of Michigan, E1, Part B).
- Each group but alternating students should do three pH measurements & test acid strength using 2 Mg strips/group.

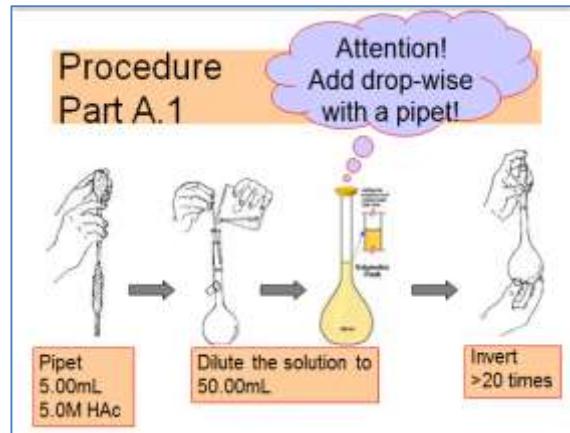
Chemicals used	Materials used
0.50 M HCl 5.0 M HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> Unknown CH <sub>3</sub> COOH solutions Magnesium wire strips or shavings	pH Meter Well plate

1. Pipet 5.00 mL of 5.0 M CH<sub>3</sub>COOH solution into 50-mL volumetric flask. Dilute the solution by filling the volumetric flask with de-ionized water until the meniscus reach the mark. Then pour 20.0 mL of this solution into a 50-mL beaker.

2. Pour 20.0 mL of 0.50 M HCl solution into another 50-mL beaker.

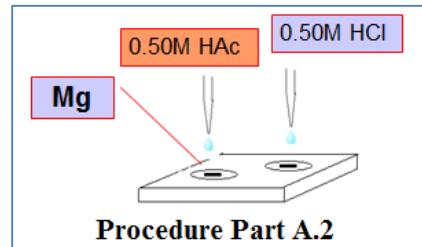
3. Pour 20-30 mL of the unknown solution CH<sub>3</sub>COOH into another third 50-mL beaker.

4. Using a calibrated pH meter, measure the pH of the solutions inside each of the three beakers. Record the measured pH.



## PART A.2: Reaction of Mg Metal with Addition of Strong and Weak Acids

5. Clean surface of two short Mg strips by lightly sand with a piece of abrasive paper and put each in the bottom of the 2 empty wells of a porcelain spot plate, add 5-10 drops 0.50 M HCl solution to one well & equal amount of CH<sub>3</sub>COOH solution to the other well just to cover the Mg strip. Record your observations.



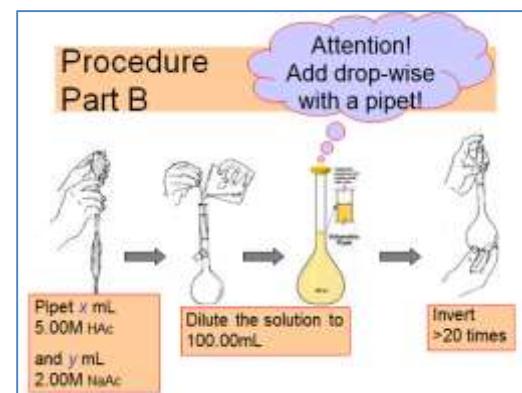
## Part B: Designing a Buffer Solution

- Each student of a group will design and prepare one pH of the buffer (four pH buffer solutions / group)

Chemical used	Materials used
5.0M CH <sub>3</sub> COOH	Graduated pipet and bulb
2.00M CH <sub>3</sub> COONa	100- mL Volumetric flask Plastic Pasteur pipet

### Design each buffer

1. You will be assigned to prepare CH<sub>3</sub>COOH / CH<sub>3</sub>COONa buffer solutions. A good first step is to look up the value of the K<sub>a</sub> for acetic acid and then calculate its pK<sub>a</sub>. For example propanoic acid, K<sub>a</sub>=1.3×10<sup>-5</sup> at 25°C or pK<sub>a</sub> = 4.89. Make buffers with 5.0 M CH<sub>3</sub>COOH and 2.0M CH<sub>3</sub>COONa solutions. Calculate the appropriate volumes x & y to be used and make the buffers next.
2. Prepare 0.100L of the buffer in a 100-mL volumetric flask: Pipet (x) mL of 5.0M CH<sub>3</sub>COOH and (y) mL of 2.00M CH<sub>3</sub>COONa solution into a 100mL volumetric flask. Dilute the solution by filling the volumetric flask with de-ionized water until the meniscus reach the mark. **Be sure to save your buffers for Procedure Part D.**



**Note:** Only prepare the following 4 buffers, one per each student, 4 buffers per group. Each student in a group tests one type of buffer all the way to end of the experiment while the other students of the group test the remaining buffers.

3. Follow the sample calculations next to design the buffer and record your data in the following table:

Buffer 1—0.100L of a pH=4.15 buffer with a total concentration of 0.100M

Buffer 2—0.100L of a pH=4.75 buffer with a total concentration of 0.100M

Buffer 3—0.100L of a pH=4.57 buffer with a total concentration of 0.100M

Buffer 4—0.100L of a pH=5.35 buffer with a total concentration of 0.100M

Buffer	Design pH	c <sub>b</sub> /c <sub>a</sub>	x mL (5.00M CH <sub>3</sub> COOH)	y mL (2.00M CH <sub>3</sub> COONa)
Example	4.00	0.178	1.7	0.75
1	4.15			
2	4.57			
3	4.75			
4	5.35			

## Sample calculations of x & y

Design the buffer CH<sub>3</sub>COOH / CH<sub>3</sub>COONa with desired pH = 4.0, where CH<sub>3</sub>COONa is the conjugate base of the weak acid CH<sub>3</sub>COOH with concentrations of C<sub>b</sub> = [CH<sub>3</sub>COONa] & C<sub>a</sub> = [CH<sub>3</sub>COOH]. Then in general, the Henderson – Hasselbalch equation for any conjugate acid–base pair can be written: (K<sub>a</sub> = 1.85 x 10<sup>-5</sup>, or pK<sub>a</sub> = 4.75 at 25 °C)

$$pH = pK_a + \log \frac{[base]}{[acid]}$$

$$\text{or } C_a/C_b = 10^{(pH-pK_a)} = 10^{(4.00 - 4.75)} = 0.178 = C_b/C_a$$

$$\text{or } C_b = 0.178C_a, \text{ but } C_a + C_b = 0.100 \text{ M}$$

Then C<sub>a</sub> = 0.085M & C<sub>b</sub> = 0.015M, but because of dilution in step 1:

5.00 M x V<sub>a</sub> (mL) = (100 mL x 0.085 M), then V<sub>a</sub> = 1.7 mL = x

Similarly: 2.00 M x V<sub>b</sub> (mL) = 100 mL x 0.015M, V<sub>b</sub> = 0.75 mL = y

## Preparing samples from each buffer solution

4. Share your results with your group in order to complete the previous table.
5. Each student, carefully pour one of three portions of 20.0 mL of the selected buffer, into a 50-mL graduated cylinder and then transferring each of the 20.0 mL portions into a 50-mL beaker. (Note: use three 50-mL beakers of same buffer per student and alternate buffers for the remaining students so each group will have prepared 12 samples, each in its own separate 50-mL beaker).
6. Using graduated cylinder the group measure two 20.0-mL portions of distilled H<sub>2</sub>O and pour each into its own 50-mL beaker.
7. Label all beakers (3 beakers of the same buffer per student & two beakers of distilled water per group & and follow the procedure next).

### Part C: Properties of a Buffer

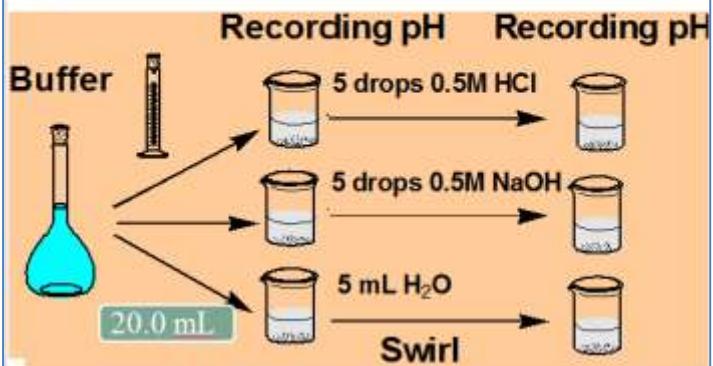
- 3 beakers/buffer/student, 12beakers/group, plus 2 beakers of distilled water/group

Chemical used	Materials used
5.0M CH <sub>3</sub> COOH	pH Meter, Funnel, Plastic Pasteur pipet
2.00M CH <sub>3</sub> COONa	Graduated pipet and bulb
0.50 M HCl	50- mL Graduated cylinder
0.50 M NaOH	100- mL Volumetric flask, 100- mL Beaker (4)

1. Each student of a group uses a pH meter to measure and record the pH of one beaker of his own buffer prepared in step 5 of Part B.. Then add 5.0 mL of deionized H<sub>2</sub>O to the same beaker and measure pH again. (two pH readings per student, 8 pH readings per group). Depending on time remaining for the experiment, instructor may ask you to skip measuring the pH before adding the 5.0 mL water in this step.

2. Each **student**, of a group uses a clean disposable pipet to add 5 drops of 0.50 M HCl to one of the two beakers containing your prepared sample buffer of step 5 Part B, and 5 drops of 0.50 M NaOH to the other beaker. Swirl the solution, measure and record the resulting pH. (two pH readings per student, 8 pH readings per group)
3. For the two beakers containing only distilled water in step 6 Part B, each **group** measures the pH of each beaker. Then with a clean, disposable pipet, add 5 drops of 0.50 M HCl to one of the beakers, and 5 drops of 0.50 M NaOH to the other beaker. Record data. Depending on time remaining for the experiment, instructor may ask you to skip measuring the pH before adding HCl or NaOH in this step.
4. Tabulate all results.

#### Procedure Part C: Properties of Buffer



#### Part D: Determination of Buffer Capacity

- One buffer capacity per students, four buffer capacities per group

Chemicals used	Materials used
0.50M HCl or 0.50 M NaOH Buffer from Part B (50 mL)	pH Meter 50- mL Graduated cylinder 100- mL, 250- mL Beakers, Funnel 50- mL Buret and buret stand

1. Fill a clean graduated pipette with 0.50M HCl solution (or use a 1-mL graduated pipet but not eye dropper). Record the initial volume  $V_1$ . Add the 0.50 M HCl solution drop-wise into the buffer with a pipet (students should use only the same buffer tested in Procedure Part C (step2) that contained 5 drops HCl), swirl the beaker after each addition. While monitoring the pH, record the volume of HCl solution to reach your buffer's capacity, record  $V_2$ . Do not exceed the buffer capacity since this may cause the need of excessive cleaning of the electrode later on.
2. Make sure you add to the volume the 5 drops of HCl already inside the buffer sample from Procedure Part C (step 2). Then exchange data with other people who had a different buffer and prepare the data sheet information in a tabulated form.



#### Procedure Part D: Buffer Capacity

## Common notes

- Buffer capacity is reached when pH starts to increase rapidly with continued addition of 0.50M HCl. This part is done by adding more drops of the HCl solution to the same beaker that you previously added 5 drops HCl. Students beware to account for the initial 5 drops volume (add volume of 5 drops from previous step Part C and the additional added volume to reach capacity).
- Each student should measure only 1 pH capacity end point, 4 end points per group. Students must use the 1mL (or as provided) graduated pipette to add all the drops in Part C & part D, then account for the volumes only for part D.
- Eye dropper is not the correct tool to add the drops for this experiment. An alternative method is to substitute the 0.50M HCl with the 0.50M NaOH but in this case you must use the corresponding beaker that you already added to it few drops of NaOH.
- Instructor/TA will give you direction if HCl or NaOH you should use to reach capacity.

## Desired data sheet information to tabulate

Discuss results & complete the post lab report for the experiment. All information in the report must be handwritten and include all the following data:

1. Buffer number.
2. Target pH, Calculated  $C_b / C_a$ .
3. Calculated  $x$  (to measure & add it to the 100mL volumetric flask).
4. Calculated  $y$  (to measure & add it to the same 100mL volumetric flask).
5. Actual pH after diluting the 100mL volumetric flask to mark (measure only once in one of the three beakers).
6. pH measured after adding 5drops 0.50M HCl to only one buffer beaker that contains 20mL buffer sample (note that total 5 beakers available : 3 buffer samples & 2 de-ionized water samples, 20 mL each sample beaker).
7. pH measured after adding 5 drops 0.50M NaOH to another beaker of the 3 buffer samples.
8. pH measured after adding 5mL de-ionized water to the third beaker of buffer sample.
9. Initial volume of 0.50M HCl before adding more drops to test buffer capacity (**Part C**).
10. Final volume after adding more drops of 0.50M HCl to capacity point.
11. Change in volume, end pH reading at capacity point.
12. Record average temperature displayed by the pH meter.

## E2: The Properties of Buffers: Resisting Change in a Turbulent World

Name:	Lab Instructor:
Date:	Lab Section:

### V. PRE-LABORATORY EXERCISES (PLE)

1. Define the term “common-ion effect” used in the BACKGROUND section. How do buffers work by taking advantage of the common-ion effect?
2. Give an example of a salt that could be used to make a buffer with NH<sub>3</sub>.
3. If solution A has pH of 3.23 and solution B has a pH of 4.23, what is their relationship in terms of [H<sup>+</sup>]?
4. What is the relation between the concentration of buffer components and the buffer capacity? Explain.
5. For a buffer having a [base]/[acid] ratio of 1:1, what is the relationship between pH and  $pK_a$ ?
6. Using Equations 1 and 2 as models, write the acid dissociation reaction and  $K_a$  value for acetic acid and calculate its  $pK_a$  value (also record these values in your laboratory notebook so you will have them available during the experiment).

## E2: The Properties of Buffer: Resisting Change in a Turbulent World

Name:	Lab Instructor:
Date:	Lab Section:

## VI. RESULTS AND POST – LABORATORY QUESTIONS (PLQ)

### Part B. Designing a Buffer

Record your entire experimental data initially on the PLQ datasheet shown by the end of this PLQ (page17). Attach a copy of your work showing the calculations of the volumes of acid and base needed to create the target buffer you were assigned.

Which set of data is yours? Buffer 1, Buffer 2, Buffer 3, or Buffer 4

	Buffer 1	Buffer 2	Buffer 3	Buffer 4
[base]/[acid] ratio				
Average pH of buffer				
Average pH of DI-H <sub>2</sub> O				
pH of buffer after adding HCl				
pH of buffer after adding NaOH				
pH of DI - H <sub>2</sub> O after adding HCl				
pH of DI - H <sub>2</sub> O after adding NaOH				

### QUESTIONS FROM PART B

1. Verify that the volumes of the acid and base you were asked to use will result in the correct total concentration of the buffer.
2. Calculate the [base]/[acid] ratio used for your buffer.

- Closely examine the data from Part B, looking at all four buffer solutions, do you see a relationship between the  $pK_a$  value of the acid, the desired or target pH of the buffer, and the [base]/[acid] ratio? Explain.
- How does the relationship in question 3 help you decide which conjugate acid–base pair to choose when designing a buffer?
- After performing Part C, did you notice a difference between the buffer and the DI–water when the strong acid/base was added? Did the buffer “do its job”? Explain.
- After performing part C with the buffer, did you notice that the designed pH value is different than the measured pH of a buffer? Explain factors that may cause difference.

#### **Part D. Determination of Buffer Capacity**

Which set of data is yours? Buffer 1, Buffer 2, Buffer 3, or Buffer 4.

	Buffer 1	Buffer 2	Buffer3	Buffer 4
Conjugate acid-base pair used				
Moles of acid in buffer				
Total volume of NaOH to reach buffer capacity				
Total moles of NaOH to reach buffer capacity				

How does the total moles of NaOH needed to reach buffer capacity relate to the number of moles of acid in the buffer? Explain.

**PLQ Datasheet: Record all your experimental data on this sheet and submit with your report. Include your group members' data (rows 11-14).**

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
<b>VC211 DATASHEET FOR EXPERIMENT: E2 PROPERTIES OF BUFFERS</b>																			
SECTION:		LAB ROOM:																	
1	2	TA <sub>1</sub>																	PAGE 1
3	4	PART B: TA INSPECTS X,Y TABLE PART B																	"pH Design"
5	6	PROEDURE PART →	A	A1	A1	A1	A2	A2	C	C	C	C	C	C	C	C	C	D	
7	8	pH METER EFFORTS	→	PH CALIB. STOCK	HCl	HAc	MS	MS	Design Buffer	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	→ BUFFER pH: H <sub>2</sub> O, NaOH, & HCl →							
9	10	Avg. pH meter Temp. = °C		0.50M	0.50M	?M	0.50M	0.50M	Buffer	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	INIT. H <sub>2</sub> O NaOH	TOT. mL	TOT. mL	TOT. mL	TOT. mL	TOT. mL	TOT. mL	
11	12	GRP NAME	#	Chinesse	ID	pH	pH1	pH2	Buffer	pH4	pH5	pH6	pH7	pH8	pH9	pH10	pH11	V(HCl)	
13	14	See B2 PPT for clear instr	20mL No Dilute Dil.	20mL 5.0mL in	20mL 5.0mL	MS	Fast/S Low Dil.	MS	Fast/S Slow RXN	MS	Fast/S Slow RXN	MS	Fast/S Slow RXN	MS	Fast/S Slow RXN	MS	Fast/S Slow RXN	MS	
15	16	Record above your name pH meter Avg. Temp.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	flask Vol.	
17	18	Grad pipet (use to deivr same the 5 grad drops pipet.)																	
19	20	Follow E2 proc.																	
21	22																		
23	24																		

# SAMPLE DATASHEET FOR A LAB SECTION

## (FOR REFERENCE ONLY, STUDENTS TO OMIT THIS PAGE)

VC211 DATASHEET FOR EXPERIMENT: E2 PROPERTIES OF BUFFERS																			PAGE 1	
SECTION:		TA:				LAB ROOM:														
BUFFER DESIGN: <b>PART B</b>		#1	#2	#3	#4	Start with (x) mL 5.0 M HAc & (y) mL 2.0 M AcNa. TA will compare "pH Design" to measured "pH8" of each buffer made by a student														
		pH	4.15	4.57	4.75	5.35														
PROCEDURE PART→		<b>A</b>	A1	A1	A1	A2	<b>C</b>	C	C	C	C	C	C	C	C	C	D	D		
EFFORTS		<b>pH METER CALIB BUFFER STOCK</b>	← —— GROUP EFFORTS —— →				<b>INDIV.</b>	<b>Group efforts: H<sub>2</sub>O only</b>												
GRP	NAME	ID	HCl	HAc	THAc	Mg	Mg	Design Buffer	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O / H <sub>2</sub> O /	BUFFER pH: H <sub>2</sub> O, NaOH, & HCl →	HCl	INIT.	H <sub>2</sub> O	NaOH	HCl	HCl	TOT. mL
#	Chinese		0.50M	0.50M	2M	0.50M	0.50M	pH	pH4	pH5	pH6	pH7	pH8	pH9	pH10	pH11	pH12	V(HCl)		
		pH	pH1	pH2	pH3	HC1	HAc													
11	1	E2 SUMMARY SUPPLEMENTS E2 DATASHEET						4.15												
12	1	Henderson-Hasselbalch equation: Showed in VC210																		
13	1	pKa = pH + log (C <sub>b</sub> /C <sub>a</sub> ), C <sub>b</sub> & C <sub>a</sub> for weak acids at equilibrium same as initial																		
14	1	Prepare Buffer for E2: Example pH = 4.75 + log C <sub>b</sub> /C <sub>a</sub> & C <sub>b</sub> + C <sub>a</sub> = 0.10, 2 Eq. 2 unknowns yield:																		
15	2	C <sub>a</sub> = 0.085M & C <sub>b</sub> = 0.015M for pH = 4.0. Now from C1V1 = C2V2 for dilution, then V1=C2V2/C1 or for:																		
16	2	V <sub>A1</sub> =V <sub>A2</sub> × C <sub>A2</sub> /C <sub>A1</sub> or V <sub>A1</sub> = 100mL × 0.085/5.0=1.7mL, & V <sub>B1</sub> = V <sub>B2</sub> × C <sub>B2</sub> /C <sub>B1</sub> = 100mL × 0.015/2.0=0.75mL																		
17	2	(best to follow datasheet after pH meter calibration)																		
18	3	1. <b>PART A:</b> Measure pH of: 0.50M HCl, 0.50M HAc (diluted from 5.0M HAc), and unknown molarity of HAc sample. Then test their strength in dissolving Mg strip. (3 pH measurements & 2 Mg strips tested /group). <b>GROUP EFFORTS</b>																		
19	3	2. <b>PART B:</b> Design 4 buffers with different pH using same stock solutions of HAc & AcNa but each buffer is diluted to 100 mL (use different volumes of each stock that will be calculated to give designed pH). (1buffer/student, 4buffers/group)																		
20	3	3. <b>PART C:</b> Test the initial pH of each buffer to compare with target pH. Then add 5 drops of 0.50M HCl to one 20mL buffer sample then add 5 drops of 0.50M NaOH to another 20mL sample of same buffer. Add 5ml de-ionized water to the 3 <sup>rd</sup> beaker of 20mL sample of same buffer. Measure pH to conclude that pH remains about constant. Measure pH for the remaining 2 water beakers of 20mL distilled water before and after using 5drops of 0.5M HCl for one beaker & 5drops 0.50M NaOH for the other beaker. If only 100-mL beakers available then use 30mL samples. Note: Datasheet pH columns not in order as procedure.																		
21	4	4. <b>PART D:</b> Test the total capacity of a buffer using the previous sample from Part C when 5drops of 0.50M HCl were added (not NaOH) by also adding more drops until pH starts dropping rapidly. Make sure to account for the initial drops of 0.50M HCl added at start of Part C, to find volume added to capacity. (1 buffer sample/student, 4buffer samples/group).																		
22	4	Data Errors: not following procedure, temperature effect on K <sub>sp</sub> contamination, pH meter errors & mishandling (electrode saturated), stock solutions, readings & dilutions, etc.																		
23	5																			
24	5																			
25	5																			
26	5																			
27	5																			
28	5																			
29	5																			
30	4																			
31	5																			
32	5																			
33	5																			
34	5																			
35																				

### SUMMARY OF E2 Properties of Buffers

- PART A:** Measure pH of 0.50M HCl & 0.50M HAc (diluted from 5.0M), then test their strength in dissolving Mg strip. (3 pH measurements (pH1-pH3) & 2 Mg strips tested /group)
- PART B:** Design 4 buffers with different pH using same stock solutions of HAc & AcNa diluted to 100 mL each, but using different volumes that will be calculated. (1buffer/student, 4buffers/group)
- PART C:** Test the initial pH of each buffer to compare with target pH. Then add 5 drops of 0.50M HCl to one 20mL buffer sample then add 5 drops of 0.50M NaOH to another 20mL sample of same buffer. Add 5ml de-ionized water to the 3<sup>rd</sup> beaker of 20mL sample of same buffer. Measure pH to conclude that pH remains about constant. Measure pH for the remaining 2 water beakers of 20mL distilled water before and after using 5drops of 0.5M HCl for one beaker & 5drops 0.50M NaOH for the other beaker. (pH4-pH9) (3beakers/buffer/student, 12beakers/group, plus 2beakers of distilled water/group). Note: Datasheet pH columns are not in order of the procedure steps for Part C.
- PART D:** Test the total capacity of a buffer using the previous sample from Part C when 5drops of 0.50M HCl were added (not NaOH) by also adding more drops until pH starts dropping rapidly. Make sure to account for the initial drops of 0.50M HCl added at start of Part C, to find volume added to capacity. (pH10-pH13) (1 buffer sample/student, 4buffer samples/group)

### TABULATED DATASHEET

(pH meter calibration with buffer pH = 4.003 or as provided)

- pH meter calibration (see instructions later): Use standard buffer of pH ~4.003 or as available. In all of measurements measure 30 mL desired solution and pour into 50-mL beaker so height of liquid is enough to cover sensor of pH meter electrode. If 100-mL beaker is used then carefully tilt beaker to cover electrode.
- Prepare dilution of 5.0 M to 0.50 M in a 100-mL volumetric flask, measure pH1, pH2, & pH3 of each of: 0.50M HCl, 0.50M HAc, 2M HAc, of unknown concentration. Mg observation: two Mg strips add 5-10 drops 0.50M HCl to one strip and then 0.50M HAc to the other.
- Prepare Buffers Target pH: Target pH: Calculated C<sub>b</sub>/C<sub>a</sub>, Calculated V<sub>t</sub> (to measure & add to 100mL volumetric flask), Calculated V<sub>d</sub> (to measure & add to the same 100mL volumetric flask), pH4&pH5 of distilled water in 2 separate beakers, pH6&pH7: Add 5drops of 0.50M HCl one beaker, and 5 drops of 0.50M NaOH to the other beaker.
- pH8,pH9: measure pH of 30 mL buffer in a 50-mL beaker, then add 5 mL distilled water and measure pH again, pH10&pH11: Using same stock buffer pour measured 30mL each into two separate beakers and measure the pH after adding 5 drops of 0.5M NaOH to one beaker and another 5 drops of 0.5M HCl to another beaker, pH12: add another 5 drops of the 0.5M HCl into the last beaker that of pH11 then measure the pH12 (keep track of the initial volume of 0.50M HCl) before adding more drops to test buffer capacity (Part C), final volume after adding more drops of 0.5M HCl to capacity point, change in volume at capacity point, and pH reading at capacity point. Make enough measurements to conclude when buffer capacity is reached within a reasonable time before end of lab session. Instructor/TA will give you directions if you should use HCl or NaOH to reach buffer capacity. Note: Datasheet pH columns are not in the same order as Procedure Part C.
- Discuss results & complete their lab report for the experiment. All information in the ALR report must be completed as instructed in manual.
- Incomplete Data Errors: not following procedure, temperature effect on K<sub>sp</sub> contamination, pH meter errors & mishandling (electrode saturated), stock solutions, inaccurate readings & dilutions, etc.



## EXPERIMENT E3

### Spectrophotometric Analysis: Phosphates in Water

Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department

(Modified version of Kristen Spotz and University of Michigan General Chemistry

Laboratory Manual)

#### I. OBJECTIVES

- Practice calculating and performing dilutions of solutions.
- Determine the concentration of phosphate in a water sample by spectrophotometric analysis (see Figure 1 spectrophotometry illustration).
- Construct and utilize a calibration curve.
- Explore the dynamics of working with a larger group of students.

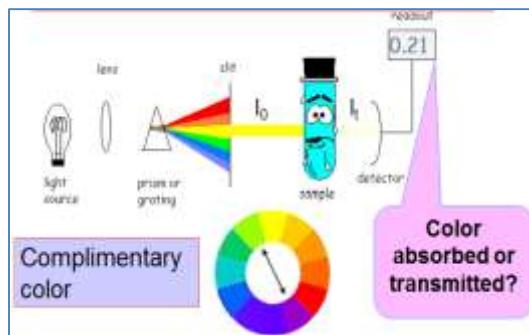


Figure 1: Spectrophotometric analysis illustration

#### II. INTRODUCTION

Imagine a time when the lakes and rivers are no longer safe for swimming or boating, or when the ocean is no longer a source of food. Coastal zones and estuaries, some of the most productive ecosystems in the world, are in danger. The problem of eutrophication, is affecting the water supply of towns across the nation making the water unsafe for consumption and hazardous to the wildlife that depend upon it.

The source of the eutrophication problem is an excessive input of nutrients into rivers, lakes and the seas because of the extensive use of fertilizers, the combustion of fossil fuels and waste from animal feedlots. This excessive nutrient input stimulates the growth of algae and bacteria, robbing the water of precious oxygen. The resulting algal blooms, red tides and deterioration of sea grass makes the waters uninhabitable for most fish and coastal wildlife.

What role will you play as a future scientist' or citizen in ensuring the protection of our valuable water resource?

#### III. BACKGROUND

Phosphates are one of the major groups of contaminants affecting our nation's water supply. Phosphates are found in environment, not only in the form have you seen in your chemistry book ( $\text{PO}_4^{3-}$ ), but also as polyphosphates (such as  $\text{P}_2\text{O}_7^{4-}$  or  $\text{P}_3\text{O}_{10}^{5-}$ ) or as organic phosphates which are eventually hydrolyzed to form  $\text{PO}_4^{3-}$ . The primary means by which humans introduce phosphates into the environment is through the use of fertilizers and detergents. In particular, tripolyphosphates ( $\text{P}_3\text{O}_{10}^{5-}$ ) have been used in soaps and detergents to combat the problem of hard water. Phosphates are also a major component of fertilizers, because phosphorus is a necessary plant nutrient and is crucial for seed formation, root development, and crop maturation. These phosphates eventually enter the water supply leaving lakes, rivers, and seas with an abnormally high phosphate concentration.

## A. Spectrophotometric Analysis and the Determination of Phosphate

Spectrophotometric analysis relies on the fact that the amount of light absorbed by a sample shows a linear dependence upon the concentration of the compound present in the solution. You have probably seen this phenomenon for yourself before. Just hold up two glasses of juice made from powdered concentrate; one made with three scoops and one made with one scoop. The more concentrated drink absorbs more light and is darker (see Figure 2). The problem with using spectrophotometric analysis in our case is that phosphates are colorless and therefore do not absorb light in the visible portion of the electromagnetic spectrum. However, due to the reactive nature of phosphates, one can easily color them using an ammonium vanadomolybdate reagent. This reagent includes ammonium metavanadate ( $\text{NH}_4\text{VO}_3$ ) and molybdate ( $\text{MoO}_4^{2-}$ ) and reacts with the phosphate to form a yellow compound (called “heteropoly acid” from here on). The formula of the yellow compound is uncertain but thought to be  $(\text{NH}_4)_3\text{PO}_4 \cdot \text{NH}_4\text{VO}_3 \cdot 16\text{MoO}_3$ . The brightness of the resulting yellow solution is directly proportional to the concentration of phosphate in the water.

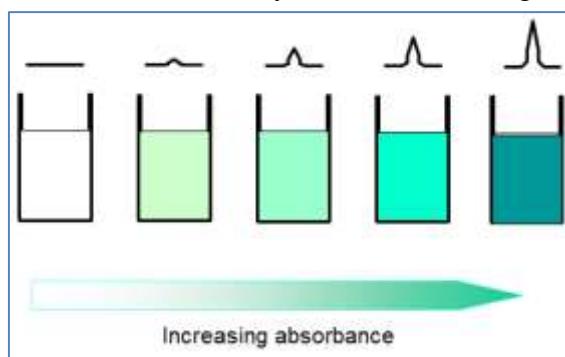


Figure 2. Concentrated solutions absorb more light

Scientists use an instrument called a spectrometer to quantitatively determine the amount of light absorbed by a solution. The primary inner parts of a typical spectrometer are illustrated in Figure 3. The spectrometer has a light source that emits light which is focused with a small slit. The wavelength of interest is then selected using the monochromator (“mono” meaning one and “chromate” meaning color) and an additional slit. The selected light then reaches the sample and depending on how the photons interact with the compound of interest, the light is either absorbed or passes straight through. By comparing the amount of light entering the sample ( $I_0$ ) with the amount of light reaching the detector ( $I$ ), the spectrometer is able to tell how much light is absorbed.

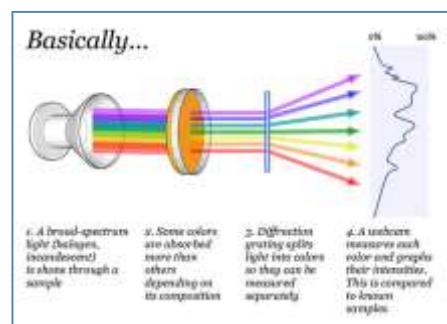


Figure 3. Working principle of spectrometer

Scientists quantify the amount of light passing through the sample in terms of percent transmittance (%T). Using Beer-Lambert law, percent transmittance is calculated as the fraction of original light intensity ( $I / I_0$ ) that passes through a sample (Equation 1).

$$\%T = \left( \frac{I}{I_0} \right) \times 100 \quad \text{Equation 1}$$

Equation 2 shows how percent transmittance (%T) can easily be converted into a quantity known as absorbance (A). Though most spectrophotometers give readings in terms of both %T and A, measurements should be made in %T and mathematically converted to A because %T can be determined more accurately.

$$A = -\log_{10}\left(\frac{\%T}{100}\right) \quad \text{or} \quad A = -\log_{10}(\%T/100) = 2 - \log_{10}(\%T)$$
Equation 2

The absorbance of a sample is important because of the previously mentioned linear relationship between absorbance and the concentration of the sample. This relationship is known as Beer-Lambert law (Equation 3).

$$A = \epsilon bc$$
Equation 3

The amount of the light that is absorbed depends on several variables (see Figure 4):

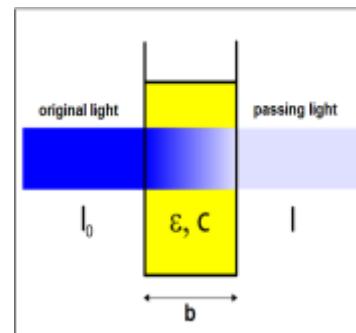


Figure 4. Light absorbance Beer-Lambert law

- “A” is the absorbance of the sample, which in this experiment is due to the interaction of phosphate, in the form of heteropoly acid, with the photons of light. Although the compound being studied may, in general, absorb light over a fairly broad range of wavelengths, there is only one region where the light is absorbed most strongly. This wavelength is known as  $\lambda_{\text{opt}}$  (pronounced “lambda optimum”). The absorbance of the sample should be measured at this wavelength to optimum reading (see illustration in Figure 8). Erratic absorbance readings may be observed if meter wave length is set near the bottom of a curve of absorbance vs. wavelength. In this case, it is recommended to change the spectrometer to a better detection method.
- “ $\epsilon$ ” is the molar absorptivity with units  $\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ . The molar absorptivity is a constant representing the efficiency by which the substance absorbs light. The greater the value of “ $\epsilon$ ” the more strongly the substance absorbs light resulting in a more intense color.
- “ $b$ ” represents the solution path length. It is the distance that the light must travel through the sample and is measured as the width of the sample holder (also called a cuvette). “ $b$ ” is a constant for each experiment (typically 1 cm).
- “ $c$ ” represents the molar concentration of absorbing species in the sample ( $\text{mol/L}$ ).

One can easily determine the unknown concentration of a sample from Equation 3 after measuring the absorbance of the sample and using the molar absorptivity of the compound and the path length of cuvette. If the molar absorptivity of the compound is not known, the concentration of an unknown can still be found by constructing a calibration curve.

## B. The calibration Curve

A calibration curve allows scientists to determine the unknown concentration of a known species.

According to Beer - Lambert law, as long as we account for a blank solution in our studies, a plot of absorbance versus concentration gives a straight line with slope = “ $\epsilon b$ ” and y-intercept

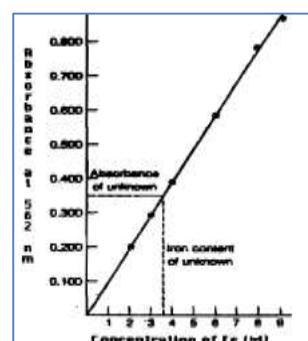


Figure 5. Fe(II) sample absorbance @562nm

= 0. For example, the calibration curve in Figure 5 is used to determine the concentration of an unknown solution of iron. The graph is constructed from six points that are made from a stock solution of iron having a known concentration. The experimentally measured absorbance of each of the six solutions is then plotted as a function of concentration and a line best fit is drawn through the points. As expected, the absorbance of the sample increases linearly as the concentration increases. The absorbance value of 0.357 was then measured for the unknown iron solution of interest. To relate the absorbance to the unknown concentration we can either use the equation of the line of best fit or we can extrapolate from the graph (as shown in Figure 5). This absorbance value was found to correspond to a concentration of 3.59 M of iron in the unknown sample.

The calibration curve in Figure 5 is an example of a successfully constructed graph. The title is labeled above the graph with both the axes clearly labeled using the independent (x-axis) and the dependent (y-axis) variables in the experiment. After plotting each of the data points on the graph, a line of best fit is drawn. Although, the points do not have to fall directly on the line, a good agreement is expected and needed for accurate determination of the concentration of your unknown.

## C. OVERVIEW

In this experiment, students will work in groups to first prepare a series of six standard solutions of known phosphate concentration by dilution of a stock solution. Using  $\lambda_{\text{max}}$  of 400nm, the absorbance of the five standard phosphate solutions will be measured and used to construct a calibration curve. The absorbance of a sample of unknown phosphate concentration will then be determined. The calibration curve will be used to relate the absorbance to the unknown concentration of phosphate in sample.

## IV. EXPERIMENTAL PROCEDURES

**"Make sure you take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5. Also closely review the lecture presentation for this E3 as posted on Canvas"**

### Part A. Preparation of Standard Solutions

Chemicals used	Materials used
Phosphate stock solution ( $1.00 \times 10^{-3}$ M) 2M HNO <sub>3</sub> Ammonium vanadomolybdate (AV) solution Two water samples (A & B) of unknown phosphate concentration	Spectrophotometer 50- mL Volumetric flask 1, 2, 5- mL Pipets and pipet bulb Cuvettes (1 per group of students) 500- mL Beakers

- Students will work in groups as assigned to construct a single calibration curve consisting of 6 data points having phosphate concentrations in the range  $2.00 \times 10^{-5}$  M to  $1.00 \times 10^{-4}$  M. Each student will be responsible for making at least one of the solutions (see Figure 6 demonstration) and measuring the absorbance of at least one data point.

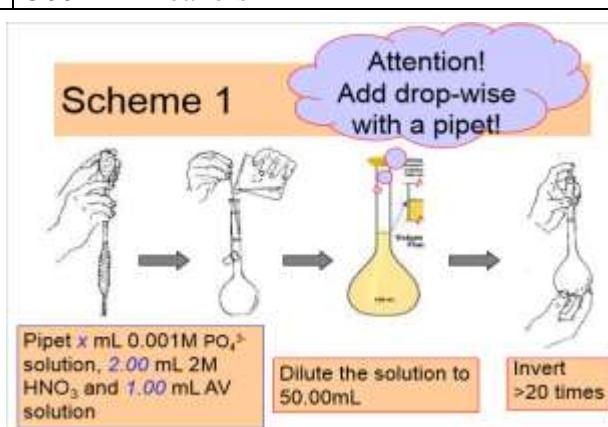


Figure 6. Dilution demonstration.

Show your instructor your calculations for making your 50- mL standard solutions from the  $1.00 \times 10^{-3}$  M phosphate stock solution before your group goes on part B. Remember, solutions must be made using only the available volumetric flask and pipets. Glassware including pipets and volumetric flasks must be washed & rinsed with distilled water as instructed. **Do not contaminate reagent bottles and follow the instructor's procedure.**

Make the following data sheet to record your data:

Sample	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>	4 <sup>#</sup>	5 <sup>#</sup>	6 <sup>#</sup>	Unknown 7 <sup>#</sup> A	Unknown 7 <sup>#</sup> B
V(mL)	0.00	1.00	2.00	3.00	4.00	5.00		
Conc.(M)								
Absorb. A								

- Based on your calculation from **step 1**, pipet 1.00mL of  $1.00 \times 10^{-3}$  M phosphate stock solution, , into a 50-mL the volumetric flask labelled as 2<sup>#</sup>.
- Pipet 2.00 mL 2M HNO<sub>3</sub> solution into the same volumetric flask.
- Pipet 1.00 mL of the ammonium vanadomolybdate (AV) stock solution into the same flask.
- Dilute the stock solution by filling the volumetric flask until the meniscus reaches the mark (Figure 7).
- Repeat steps 2 to 5 for each of the six standard solutions 2<sup>#</sup> - 6<sup>#</sup> but pipet the corresponding phosphate volume from the table instead of that shown by Step 2.
- For the preparing the blank sample 1<sup>#</sup> repeat steps 3 through 5 (skip step 2 so do not add any phosphate stock).

**Now you have prepared six standard solutions 1<sup>#</sup> through 6<sup>#</sup>, complete their corresponding data in above data sheet.**



Figure 7. Proper dilution to meniscus

### Procedure Part B. Adjusting the Spectrophotometer

1. Turn on the spectrometer (Figure 8) by rotating the power control clockwise. Allow the spectrophotometer to warm-up for 10 minutes before using. Press the “MODE” button to select %T (transmittance mode). The current mode appears on the display.

2. Adjust the wavelength to 400 nm. With no sample in the spectrometer (use only the black block and do not insert any cuvette), press the 0%T button so the meter reads 0%. Each member of the group should verify all readings.

3. *Always use the same cuvette and rinse with a few mLs of solution whenever you are using a new solution.* Discard the rinsing solution according to your instructor's directions. Three-quarters fill the rinsed cuvette with the blank solution from the standard



Figure 8. Typical spectrophotometer

sample labeled 1#. Insert the cuvette into the sample holder of the spectrometer and press the 100% T button so the meter reads 100%. Use always the same slot of the sample holder for the remains of the experiment. Your instrument is now calibrated until completion of the experiment properly.

Attention: If wavelength is changed for any reason, then you may have to calibrate the meter in transmittance setting again at either 0% or 100% mode (more quick to use 0% mode with the black block to prevent light transmittance. Check with instructor.

### Procedure Part C. Making the Absorbance Spectrum & Finding $\lambda_{OPT}$ using a Standard Solution

- Rinse the same cuvette you used for your blank with about 1 mL of your standard solution 6#. Three-quarters fill the rinsed cuvette with the sample solution. Insert the cuvette into the spectrometer. Measure and record the percent absorbance A in the range of 400-450nm but measure the data at every 10nm increments.

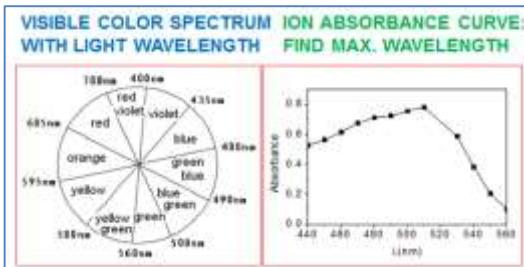


Figure 9. Determining  $\lambda_{OPT}$ .

- Record your data on the table and the datasheet. Find  $\lambda_{OPT}$  corresponding to the maximum A (similar to data in Figure 9). All data points for a given curve must be measured with the same cuvette. All phosphate solutions should be discarded according to your instructor's directions. Diagram here shows typical absorbance with wave length using same sample solution. **Your curve may look different than that shown by Figure 9.**

$\lambda$ (nm)	400	410	430	450
A				

#### Important Notes: Maximum Absorbance Optimum Wavelength ( $\lambda_{opt}$ )

- After calibration in Procedure Part B, remove cuvette and rinse in distilled water followed by sample 6# solution. Must use same cuvette, then change mode to absorbance.
- Insert a 3/4 full of sample 6# solution into the same slot as during calibration with  $\lambda = 400\text{nm}$ , then read absorbance (make sure knob is pulled to the location slot of the cuvette).
- Find the optimum wavelength when maximum absorbance is observed by varying  $\lambda=400\text{nm}-450\text{nm}$ , always increase knob in same direction and do not go backward until this Procedure Part C is completed. Do not re-calibrate the meter when changing wavelengths.

Important note: the spectrometers in the chemistry building are operating near the low ends of the Gaussians distribution curve of  $a$  vs  $\lambda$ , so higher than 400nm will produce erroneous absorbance data that can go up and down but higher than that at 400nm. Therefore,  $\lambda_{opt} = 400\text{nm}$  and the remaining absorbance data can be ignored.

Many factors can effect this erratic behavior such as: meter light sensitivity near the bottom of the Gaussians curve, accuracy of wavelength dial mechanism, accuracy of

dial reading, cuvette condition such as rinsing, cleaning, bubbles, touching, solution reproduction etc.

**Additional notes:**

- a. Make sure to use same cuvette and same slot inside spectrometer compartment
- b. Before measuring, rinse cuvette with distilled water 2-3 times and then followed with Sample 6# again 2-3 times.
- c. Only handle cuvette with the opaque side and wipe dry before inserting into spectrometer.

#### **Procedure Part D. Making the Calibration Curve Using the standard Solutions**

All data points for a given curve must be measured with the same cuvette using the same spectrometer. Before using any glassware with each new solution, the glassware must be rinsed with de-ionized water and about 1 mL of the new solution and as instructed during the lab.

1. Rinse the same cuvette you used for your blank with about 1 mL of your standard solution 2#. Three-quarters fill the rinsed cuvette with the sample solution.
2. Insert the cuvette into the spectrometer. Measure and record the percent absorbance A in the range at  $\lambda_{OPT}$  once.
3. Repeat step 1 for the standard samples solutions 3# through 5# but only measure 1 absorbance for each sample 2# - 5#.
4. Record your absorbance data on the table shown by procedure Part A & on the datasheet.

#### **Procedure Part E. Determination of Unknown Concentration**

1. Pipet 5.00 mL of the unknown phosphate solution, 2.00 mL of **2M** HNO<sub>3</sub> and 1.00 mL of the ammonium vanadomolybdate (AV) solution into the 7# 50-mL volumetric flask.
2. Dilute the solution by filling the volumetric flask until the liquid reaches the meniscus mark.
3.  $\frac{3}{4}$  fill the rinsed cuvette with the unknown solution. This is Unknown 7# A. Use the spectrometer to measure its absorbance A. Using solution and same cuvette (empty), repeat procedure here to make Unknown 7# B and then measure its absorbance.
4. Now determine the unknown concentration by using the calibration curve (for further information, see references at the end of this experiment).
5. **Before you leave**, make sure everyone in your group has recorded on the datasheet, the concentration and the absorbance A for each of the various phosphate solutions. *Please make the curves by using software such as origin or excel.*

## E3: Spectrophotometric Analysis: Phosphates in Water

Name:	Lab instructor:
Date:	Lab section:

## **V. PRE-LABORATORY EXERCISE (PLE)**

1. Define the underlined words in the **BACKGROUND** section.
  2. In your own words, summarize the purpose of a calibration curve.
  3. To prepare yourself for performing the dilutions required in this laboratory experiment, read the section on dilutions in your textbook (Chapter 4, section on dilution). What volume of  $1.00 \times 10^{-3}$  M phosphate stock solution is required to make 25.0 mL of a  $4.00 \times 10^{-5}$  M solution?
  4. Using the spectrophotometer, a sample was analyzed and found to have a percent transmittance of 85%.
    - a) What percent of light was actually absorbed by the sample?
    - b) Calculate the absorbance (A) of the sample.

### **E3: Spectrophotometric Analysis: Phosphates in Water**

<b>Name:</b>	<b>Lab instructor:</b>
<b>Date:</b>	<b>Lab section:</b>

**Record your initial measured raw data on the attached datasheet for E3 (page10), then record that data on the TA datasheet before you leave the lab session. Also, you should continue reporting the measured data on your ALR report as instructed.**

## **VI. RESULTS AND POST-LABORATORY QUESTIONS (PLQ)**

1. Attach a copy of your data table from today's experiment. Your table should include the concentration of phosphate in each standard solution, the measured %T and your calculated absorbance. You may copy the table shown in the Procedure – Part A.
2. Attach a copy of your calibration curve. What is the equation of the best-fit line?
3. Determine the concentration of phosphate in your unknown solution by extrapolation of the calibration curve (refer back to Figure 2) and by using the equation for the line of best fit. The extrapolation should be shown on your attached calibration curve. The calculation using the line of the best fit should be shown below.
4. The U.S. Public Health Service has set the maximum value of phosphate in the drinking water at 0.30 mg phosphate/liter. Did your unknown water sample violate this standard? Show your work.

THE FOLLOWING DATASHEET OF YOUR RAW MEASUREMENTS SHOULD BE COMPLETED DURING THE LAB SESSION AND ATTACHED TO THE PLQ. YOU MUST BRING A COPY OF THIS SHEET TO YOUR LAB.

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S
2																		
3	<b>SECTION: TA:</b> <b>GROUP EFFORTS &amp; INDIVIDUAL REPORTS: EACH STUDENT SHOULD PREPARE AT LEAST ONE SAMPLE SOLTN. 1 CALIBRATION DATA &amp; COMPLETE ROW DATA</b>																	
4	<b>PART A: PREPARE 6 STANDARD CAL. SAMPLES + 1 UNKNOWN</b> ←----- PARTS A, D, & E; SAMPLES PREPARED WITH SHOWN VOLUMES (mL) -----→																	
5	<b>PART B: CALIBRATION @ 400nm 0% T (black block) &amp; 100% T (Sample 1#)</b> ←PART A: CALIBR. SAMPLES WITH SHOWN STAND.VOLUME SOLNS.-----→																	
6	<b>PART C: FIND λ<sub>OPT</sub> of 6# SAMPLE</b> ←PART D: ABSORBANCE OF PREPARED SAMPLES @ λ <sub>OPT</sub> -----→																	
7	<b>PART E: UNKNOWN ABSORB @ λ<sub>OPT</sub> →</b> ←PART F: UNKNOWN CONC. (M) CONC. (M) →																	
8																		
9	NAME	ID #	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12	A13	A14	CONCN CONCN	
10	GR P # →	400nm	410nm	420nm	430nm	450nm.....nm	0.00mL	2.00mL	3.00mL	4.00mL	5.00mL	5.00mL	5.00mL	5.00mL	5.00mL	5.00mL		
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18																		
19																		
20																		
21																		
22																		
23																		
24																		
25																		
26																		
27																		
28																		
29																		
30																		
31																		
32																		
33																		
34																		
35																		
36																		
37																		
38																		
39																		
40																		
41																		

EACH STUDENT MUST PREPARE AT LEAST 1 SAMPLE SOLUTION. ALL E2 IS GROUP EFFORTS EXPERIMENT BUT MUST SUBMIT INDIVIDUAL REPORT. EACH STUDENT MUST COMPLETE 1 ROW OF DATA & ENTER ON THE MAIN DATASHEET. DO ALL PARTS E3-E6

**DO NOT WASTE REAGENTS:** AV difficult to make, follow rinsing pipet instructions.

**Part A:** Preparation of 6 Standard Solutions: from  $1.00 \times 10^{-5}$  M to  $4.00 \times 10^{-4}$  M (Pipet x mL 0.001M  $\text{PO}_4^{3-}$  solution, 2.00 mL 2M  $\text{HNO}_3$  & 1.00 mL AV solution, into 50-mL volumetric flask & dilute with distilled  $\text{H}_2\text{O}$  to mark

**Part B:** Adjusting the Spectrometer @  $\lambda = 400\text{nm}$ , rinsing the cuvette

**Part C:** Finding the proper wavelength ( $\lambda_{OPT}$ ) for maximum absorbance by using standard stock that has the maximum concentration (stock # 6) and then find its maximum absorbance at varying wavelengths from 400nm to 450nm to find  $\lambda_{OPT}$

**Part D:** Making the calibration curve using the standard stock solutions (#1 to #6) at maximum absorbance ( $\lambda_{OPT}$ ) wavelength.

**Part E:** Determination of the unknown concentration by:

- a. Using projection from the standard curve.
- b. Using slope of the standard curve then Beer's law:  

$$[A = \text{slope } x c] \& A = -\log(\%T/100)]$$

Make sure to use Excel or Origin software to plot & calculate.

**TYPICAL SPECTROMETER SETUP**



6. Use Sample 6# cuvette (5mL  $\text{PO}_4^{3-}$ ). Change mode to Absorbance. Record [A1].

5. Use a blank Sample 1# cuvette (0mL  $\text{PO}_4^{3-}$ ) to adjust T = 100.0% if different than display.

4. Use a black block to adjust T = 000.0% if different than display.

3. Insert black block and the 2 cuvettes (3/4 full) into the 3 slots shown

2. Adjust the  $\lambda$  to 400nm & mode to Transmittance

1. Turn power on (15 min.)

7. Find  $\lambda_{OPT}$ : Adjust  $\lambda$  to 410nm, repeat steps 4-6. Record [A2]

8. Repeat step 7 at  $\lambda = 420-450\text{nm}$ . Record corresponding A3-A6

9. Find  $\lambda_{OPT}$  then adjust dial  $\lambda$  to  $\lambda_{OPT}$ . **Do not touch the dial any more.** Repeat steps 4-6 at  $\lambda_{OPT}$ . Record [A12].

10. Replace Sample 6# cuvette with Sample 2# cuvette (1mL 5mL  $\text{PO}_4^{3-}$ ). Repeat steps 4-6 but using the replacement cuvette instead of cuvette in Step 6. Read [A8].

11. Repeat Step 10 for the remaining samples [A9-A12 or A9-B12].

**Pull/Push knob here**

**SAMPLE DATASHEET FOR A LABORATORY SECTION**  
**(FOR REFERENCE ONLY, STUDENTS TO OMIT THIS PAGE)**

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	
5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
GR	NAME	ID #	λ1 400nm	λ2 410nm	λ3 420nm	λ4 430nm	λ5 450nm	λ <sub>OPT</sub> .....nm	0.00mL	1.00mL	2.00mL	3.00mL	4.00mL	5.00mL	5.00mL	5.00mL	5.00mL	5.00mL	
P # →	GR	ABSORBANCE (A)	A1	A2	A3	A4	A5	A6	BLANK	A7	A8	A9	A10	A11	A12	A13	A14	CONC'N	CONC'N
1																			
2		Maximum Absorbance Optimum Wavelength ( $\lambda_{opt}$ )																	
3		1. After calibration in Procedure Part B, remove cuvette and rinse in distilled water followed by sample 6# solution.																	
4		Must use same cuvette, then change mode to absorbance.																	
5		2. Insert a 3/4 full of sample 6# solution into the same slot as during calibration with $\lambda = 400\text{nm}$ , then read absorbance (make sure knob is pulled to the location slot of the cuvette).																	
6		3. Find the optimum wavelength when maximum absorbance is observed by varying $\lambda=400\text{nm}-450\text{nm}$ , always increase knob in same direction and do not go backward until this Procedure Part C is completed. Do not re-calibrate the meter when changing wavelengths.																	
7		Important note: the spectrometers in the chemistry building are operating near the low ends of the Gaussians distribution curve of a vs $\lambda$ , so higher than 400nm will produce erroneous absorbance data that can go up and down but higher that that at 400nm. Therefore, $\lambda_{opt}=400\text{nm}$ and the remaining absorbance data can be ignored. Many factors can effect this erratic behavior such as: meter light sensitivity near the bottom of the Gaussians curve, accuracy of wavelength dial mechanism, accuracy of dial reading, cuvette condition such as rinsing, cleaning, bubbles, touching, solution reproduction etc.																	
8		Additional notes:																	
9		a. Make sure to use same cuvette and same slot inside spectrometer compartment																	
10		b. Before measuring, rinse cuvette with distilled water 2-3 times and then followed with Sample 6# again 2-3 times.																	
11		c. Only handle cuvette by the opaque 2 sides and wipe dry carefully with proper tissue before inserting into its slot inside the spectrometer rack.																	
12																			
13																			
14																			
15																			
16																			
17																			
18																			
19																			
20																			
21																			
22																			
23																			
24																			
25																			
26																			
27																			
28																			
29																			
30																			
31																			
32																			
33																			
34																			
35																			
36																			
37																			
38																			
39																			
40																			
41																			
42																			
43																			
44																			

Procedure Part A: each student prepare 2 samples, group leader 1 sample  
 Procedure Part B: each student verify calibration  
 Procedures Part C, D, E: each student measure and record the remaining rows data (A1-A13(or A1-A12 & A14))  
 1. Wash & rinse all glassware.  
 2. Rinse each pipet & drive out all excess water then wipe dry the outside.  
 4 pipets available color coded: 1-mL (yellow tag for AV), 2-mL(black tag for 2M HNO<sub>3</sub>), 5-mL (red tag) for 0.001M Na<sub>3</sub>PO<sub>4</sub>, 5-mL(red tag) for unknown ?M PO<sub>4</sub><sup>3-</sup>). Keep pipets on the rack each at its original location.  
 3. Rinse pipet with stock solution (2-3 times). Minimize waste of reagents.  
 4. Using the pipet 1 mL AV stock solution into each of the 7 volumetric flasks.  
 5. Repeat step 4 by using 2mL 2M HNO<sub>3</sub>.  
 6. Repeat step 4 by pipet x mL 0.001M Na<sub>3</sub>PO<sub>4</sub> standard solution. See (x) values above. For the unknown molarity sample of PO<sub>4</sub><sup>3-</sup> dilute 5 mL in a 50.00-mL volumetric using either unknown samples A or B but not both  
 7. Dilute to 50.00mL mark  
 8. Measure absorbance of each sample at the optimum  $\lambda$ , make sure you rinse cuvette with each corresponding sample 2-3 time  
 AV = ammonium vanadomolybdate

**ADDITIONAL REFERENCES: For reference & reading only but no need to copy this section into any part of your After-Lab Report (ALR)**

## REFERENCE 1

### Doubts about E3

Sat, Mar 22, 2014 01:14 PM

Dear Professor:

I'm Michael again, and I want to ask a question on behalf of our lab team.

When we were finding the maximum wavelength corresponding to the maximum A, we had a problem which can't be solved.

First I want to show our data when carrying out Part 3:

$\lambda(\text{nm})$  400 405 410 415 420 425 430 435 440 445 450

A 0.3 0.271 0.242 0.217 0.195 0.177 0.162 0.145 0.136 0.126 0.114

As is shown above, when  $\lambda$  is 400nm, A becomes the largest. But since 400 is the smallest in the range of 400~450, we can't

determine whether A will become even larger when  $\lambda$  further decreases. Therefore, we further decreased  $\lambda$  to examine A

continuously, and we found the following data:

$\lambda(\text{nm})$  400 395 390 385...

A 0.3 0.332 0.371 0.415...

And the result is just as we expected.

So we still hold the doubt that whether we should use bigger  $\lambda$  to examine A in the following experiments? Is the range 400~450nm

too narrow for determining the  $\lambda_{\text{max}}$ ? Does this have something to do with the visible light spectrum whose range of

wavelength is 390~700nm?

Above is our current question, and I have put it in the Discussion part of my post-lab report.

Thank you so much for checking this out!

Have a nice weekend!

Michael Liu/JI Student Mar 22, 2014

Xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

## REFERENCE 2

**TO STUDENTS:** this section is for reference only & no need to copy into your report.

### FERRIC SOLUTION SPECTROPHOTOMETRIC ANALYSIS

“The phosphate calibration solution used by SJTU chemistry department (easier to prepare) has disadvantage of not having a precise  $\lambda_{\text{MAX}}$  (no peak with bell shape trend). Instead & in the future, it is preferred to use the following alternative iron solution standard (using similar experimental procedure). Fe solution displays a nice shaped bell peak for  $\lambda_{\text{MAX}}$  as shown in the reference paper at the end of this experiment.”

#### Procedure Part A. Preparation of Standard Solutions

- Pipet 0.00, 1.00, 2.00, 3.00, 4.00, 5.00mL  $20\mu\text{g}\cdot\text{mL}^{-1}$  ferric stock solution into labelled 1<sup>#</sup> through 6<sup>#</sup> 50-mL volumetric flasks, respectively. Record data in the following table and in your report.

Sample	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>	4 <sup>#</sup>	5 <sup>#</sup>	6 <sup>#</sup>	Unknown 7 <sup>#</sup> A	Unknown 7 <sup>#</sup> B
V(mL)	0.00	1.00	2.00	3.00	4.00	5.00		
Conc.(M)								
Absorb. A								

- Pipet 1.00 mL 10% hydroxylamine hydrochloride and 5.00mL 1.0M NaAc solutions into each of the labelled 1<sup>#</sup> - 6<sup>#</sup> 50 mL volumetric flasks, respectively.
- Pipet 2.00 mL Of 0.15% of Phenanthroline stock solution into each 1<sup>#</sup> - 6<sup>#</sup> 50-mL volumetric flasks.
- Dilute the solution by filling the volumetric flask until the liquid reached the meniscus mark.

#### **Procedure Part B. Adjusting the Spectrophotometer**

- Turn on the spectrometer to warm-up (15min).
- Adjust the wavelength to 440nm. Use a black block to adjust T = 0%. Do not insert any cuvette in sample holder.
- Wash and rinse the cuvettes. Insert a cuvette filled up to ¾ volume with solution from the 1<sup>#</sup> prepared solution to set T = 100%. Only insert the cuvette either on the left hand side or the right hand side space and keep using the same position throughout the entire experiment.

#### **Procedure Part C. Making the Absorbance Spectrum Using a Standard Solution**

- Rinse another cuvette and ¾ fill the rinsed cuvette with solution from the 6<sup>#</sup> prepared solution.
- Insert the cuvette into the spectrometer. Measure and record absorbance A in the range of 440-560nm but measure the data at every 5nm increments.
- Find  $\lambda_{OPT}$  corresponding to the maximum A.

#### **Procedure Part D. Making the Calibration Curve using the Standard Solution**

- All data points for a must be measured with the same cuvette using the same spectrometer
- Rinse the same cuvette, ¾ fill the rinsed cuvette with the 2<sup>#</sup> solution.
  - Insert the cuvette into the spectrometer. Measure and record A at  $\lambda_{max}$  once.
  - Repeat above step for 3<sup>#</sup> - 5<sup>#</sup> solutions but only measure 1 absorbance for each sample 2<sup>#</sup> - 5<sup>#</sup>.

#### **Procedure Part E. Determination of Unknown Concentration**

- Pipet 5.00 mL of the unknown ferric solution, Pipet 1.00 mL 10% hydroxylamine hydrochloride, 5.00mL 1.0M NaAc, and 2.00 mL Of 0.15% of Phenanthroline stock solution into the 7<sup>#</sup> 50-mL volumetric flask.
- Dilute the solution by filling the volumetric flask until the liquid reaches the meniscus mark.
- ¾ fill the rinsed cuvette with the unknown solution. Use the spectrometer to measure absorbance A.
- Now determine the unknown concentration by using the calibration curve. (for further information, see references at the end of this experiment). **Please make the curves by using software such as origin or excel.**

## REFERENCE 3

**Plot of the standard curve: showing the linear relation between light absorption and concentration of the standards**

New Delhi, February 2000

CSMRS Building, 4th Floor, Olof Palme Marg, Hauz Khas,

New Delhi – 11 00 16 India

Tel: 68 61 681 / 84 Fax: (+ 91 11) 68 61 685

E-Mail: dhvdelft@del2.vsnl.net.in

DHV Consultants BV & DELFT HYDRAULICS

with

HALCROW, TAHAL, CES, ORG & JPS

## 1. Introduction to Absorption Spectroscopy

Absorption Spectroscopic methods of analysis rank among the most widespread and powerful tools for quantitative analysis. The use of a spectrophotometer to determine the extent of absorption of various wavelengths of *visible* light by a given solution is commonly known as *colorimetry*. This method is used to determine concentrations of various chemicals which can give colours either directly or after addition of some other chemicals. As an example, in the analysis of phosphate, a reaction with orthophosphate is made, to form the highly coloured molybdenum blue compound. The light absorption of this compound can then be measured in a spectrophotometer.

Some compounds absorb light in other than the visible range of the spectrum. For example, nitrates absorb radiation of 220 nm wave length in the UV region.

## 2. Absorption Spectroscopy Theory

Absorption Spectroscopic methods of analysis are based upon the fact that compounds ABSORB light radiation of a specific wavelength. In the analysis, the amount of light radiation absorbed by a sample is measured. The light absorption is directly related to the concentration of the colored compound in the sample.

The wavelength ( $\lambda$ ) of Maximum Absorption is known for different compounds. For example, the colored compound formed for analysis of Phosphate (molybdenum blue) has maximum light absorption at  $\lambda = 640$  nm. Conversely, a minimum amount of light is transmitted through the compound at  $\lambda = 640$  nm. This is shown schematically in Figure 1.

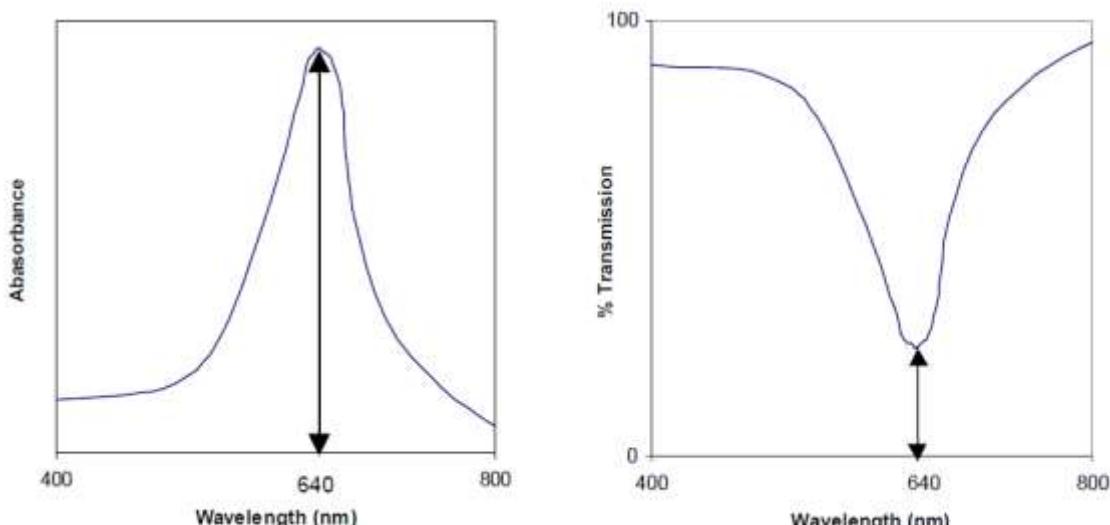


Figure 1: Light Absorption and Transmission by Phosphate-molybdenum blue compound. Schematic diagram showing maximum light absorption (and minimum light transmission) at  $\lambda= 640$  nm.

Due to the fact that the overall composition of the sample is seldom the same as that of the calibration standard, in some cases, the absorption characteristics of the two may differ. Where such discrepancy is suspected, the standard addition approach may be used. Here, a known amount of analyte is added to a second aliquot of the sample. The difference in absorbance is used to calculate the analyte concentration of the sample as illustrated in

### Example 1

A 25 mL sample after treatment with reagents to generate colour for measurement of phosphate yielded absorbance of 0.428. Addition of 1.00 mL of a solution containing 5.0 $\mu\text{g}$  phosphorus to a second 25 mL aliquot and development of colour resulted in an absorbance of 0.517. Calculate  $\mu\text{g}$  phosphorus in each mL of sample.

#### Solution:

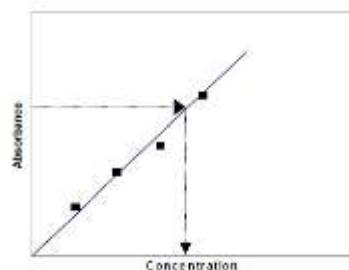
Correct absorbance for dilution:

$$\text{Corrected absorbance} = 0.517 (26.0/25.0) = 0.538$$

$$\text{Absorbance caused by } 5\mu\text{g} \text{ phosphorus} = 0.538 - 0.428 = 0.110$$

$$\text{Therefore, phosphorus in the sample} = (5.0/0.11) 0.428$$

$$= 19.5\mu\text{g}, \text{ or } 19.5/25 = 0.7\mu\text{g/mL}$$



**Figure 6: Finding the concentration of an unknown sample from the standard curve.**

### Overview of individual methods

The general procedure can be followed for all spectrophotometer analyses. For analysis of specific compounds, the method of preparation of the colored compound, and the wavelength of maximum light absorption will vary. An overview is given in Table 1.

Table 1 Overview of specific methods used for analysis of water quality parameters, and the wavelength of maximum light absorption

Parameter	$\lambda$	Method Name	SAP
Aluminum	535	Eriochrome Cyanine R Spectrophotometric	1.30
Boron	540	Curcumin Spectrophotometric	1.3
Chlorophyll a	750, 664, 65	Acetone Extraction Spectrophotometric	1.5
Flouride	570	SPADNS Spectrophotometric	1.11
Iron	510	Phenanthroline Spectrophotometric	1.13
Manganese	525	Persulphate Spectrophotometric	1.34
NH <sub>3</sub> -N	640	Phenate Spectrophotometric	1.15
NO <sub>3</sub> -N	220, 275	UV Spectrophotometric	1.16
NO <sub>2</sub> -N	543	Sulphanilamide Spectrophotometric	1.17
-PO <sub>4</sub>	880	Ascorbic Acid Spectrophotometric	1.20
Total P	880	Digestion + Ascorbic Acid Spectrophotometric	1.39
Silica	815	Ammonium Molybdate Spectrophotometric	1.38
Sulphate	420	NOTE: preferred method for sulphate analysis is with nephelometer	1.26

Spectrophotometric analysis has:

- wide applicability
- high sensitivity: detection limit 10<sup>-5</sup>M to 10<sup>-4</sup>M range
- moderate to high selectivity
- good accuracy: relative error 1 to 3%
- ease and convenience, lends to automation

## REFERENCE 4:

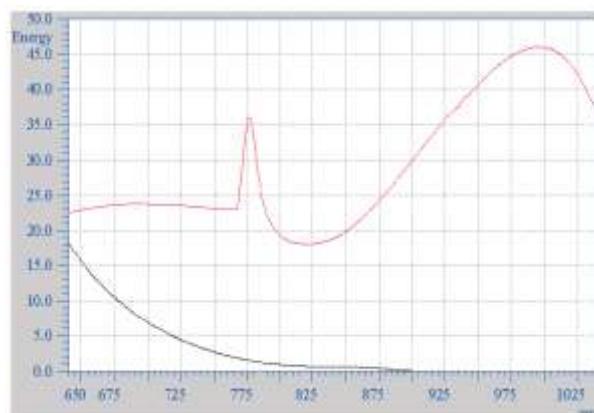


Figure 1: Comparison of the Cintra 10e raw light scan with a 200 ppb conventional spectrometer raw light scan

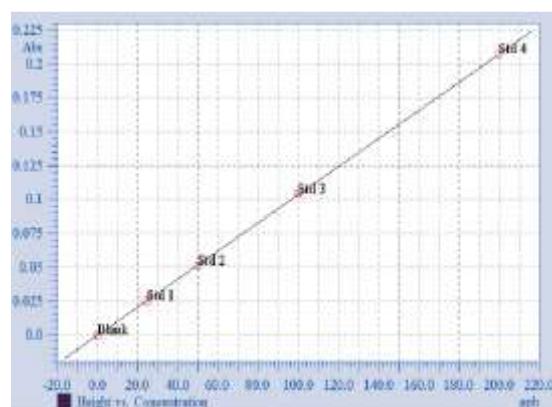


Figure 2: Calibration graph of standards from 25 to Phosphorus.

## application note

### Cintra 10e—Enhanced Sensitivity from 600–1,200 nm, Analysis of Phosphorus to Sub-ppb Levels

by Paul A. Liberatore  
Marketing Product Manager  
UV-Vis Spectroscopy

#### Introduction

The analysis of phosphorus is of importance in environmental and industrial type monitoring applications. Phosphorus has been traditionally measured in solutions using a UV-Visible Spectrometer at the compromised 470 nm or the 690 nm wavelengths<sup>1</sup>. These methods have poor detection limits and suffer from the phosphorous-complex formation taking a relatively long time to stabilize as well as producing unstable complexes.

The most sensitive 830 nm wavelength has not been commonly utilized due to very low light throughput of conventional spectrometers at this wavelength.

The reason for this is that spectrometers, that utilize a photo multiplier detector usually have an upper wavelength range of 900 nm. These instruments have very poor light throughput in the 600 to 900 nm range and hence cannot utilize the most sensitive 830 nm wavelength.

The Cintra 10e uses a Silicon Photodiode and advanced optics design, which enables a wavelength range up to 1,200 nm. The Cintra 10e also has significantly increased light throughput in the important 830 nm wavelength range.

The Molybdenum Blue phosphorous method<sup>2</sup> is a very sensitive method for the determination of phosphorus. When

this method is used in conjunction with a spectrometer with ample light throughput at 830 nm, such as the Cintra 10e, then phosphorus analyses can be determined at sub ppb concentrations. The Molybdenum Blue phosphorous method is not only easier to use than conventional methods but the blue phosphorous-molybdenum complex formed is produced very quickly and is stable for over 24 hours. This enables quick turn around time for sample analysis because long stabilization times required for complex formation (as occurs in conventional methods), are not required. As the blue phosphorous-molybdenum complex is very stable, samples do not have to be analyzed quickly, so large sample batches may be analyzed. Conventional methods produce unstable complexes, necessitating fast analysis of small sample batches.

#### Equipment

Cintra 10e  
Spectral Quantify Application  
1 cm quartz cell

For the automated analysis of up to 270 samples, use the SDS-270 auto-sampler and the auto-upper with flow through cell.

#### Cintra 10e enhanced energy above 630 nm

A Raw Light Scan is the best way to visually determine the light throughput to the detector at various wavelengths. The photomultiplier gain is kept constant and light scanned across the wavelength range. Figure 1 graphically illustrates the superiority of the Cintra 10e over conventional spectrometers in regards to light throughput above 630 nm. The Cintra 10e has a flat energy response from 630 to 760 nm, then it peaks at 770 nm drops in intensity at 800 nm and then peaks again at 1,000 nm. The energy response for a conventional spectrometer is already low at 600 nm and drops exponentially up to 900 nm.

At 830 nm the energy response of the Cintra 10e is 18.4%. Compare this to a conventional spectrometer which has an energy response at 830 nm of 0.4%. The energy response at 830 nm is nearly 50 times higher in the Cintra 10e compared to a conventional spectrometer. This increase in energy at 830 nm will result in decreased photo multiplier voltage required to amplify the signal and a

subsequent decrease in noise. The decrease in noise will also lead to improved detection limits in the case of phosphorus determination.

The increased energy at 830 nm coupled with an inherently sensitive phosphorus method enables in the measurement of sub ppb phosphorus concentrations.

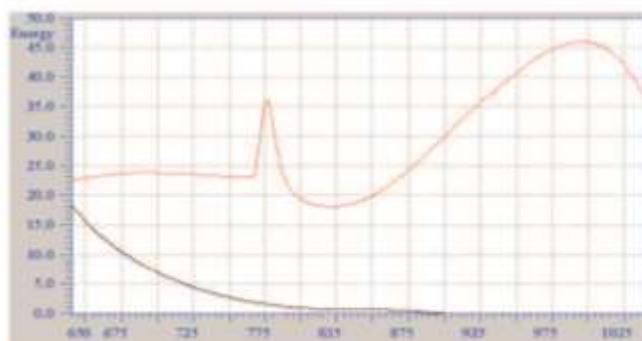


Figure 3: Comparison of the Cintra 10e raw light scan with a conventional spectrometer raw light scan

## Molybdenum Blue Method

Orthophosphate and molybdate ions condense in acidic solution to form molybdophosphoric acid (phosphomolybdc acid). Upon selective reduction, (e.g., with hydrazinium sulphate) a blue colour is produced due to Molybdenum Blue of uncertain composition. The intensity of the blue colour is proportional to the amount of phosphate initially incorporated into the heteropoly acid. If the acidity at the time of reduction is 0.5 M in sulphuric acid and hydrazinium sulphate is the reductant then the resulting blue complex exhibits a maximum absorbance at 820–830 nm.

Ions which form heteropoly acids, such as silicate, arsenate, germanium and tungstate should be absent. Silicate may be removed by fuming with perchloric acid to dehydrate the silicic acid and render it insoluble. Arsenate may be volatilized as arsenic (III) bromide from a hydrofluoric-acid sulphuric acid solution. Tin and germanium are also volatilized simultaneously.

### Reagents

#### 1. Molybdate solution.

12.5 g of Analytical Reagent sodium molybdate ( $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ ) was dissolved in 5 M sulphuric acid and diluted to 500 mL with 5 M sulphuric acid.

This solution is to be prepared fresh monthly.

#### 2. Hydrazinium sulphate solution.

1.5 g of Analytical Reagent hydrazinium sulphate was dissolved in deionised water and diluted to 1000 mL.

This solution is to be prepared fresh monthly.

#### 3. Standard Phosphate solution (10 ppm P)

0.04393 g of Analytical Reagent Potassium Dihydrogen Phosphate was dissolved in deionised water and diluted to 1000 mL 1 mL of solution = 0.01 mg P.

This solution is to be prepared fresh weekly.

#### 4. Calibration Standards

The calibration standards were prepared as per table 1. These are to be prepared fresh daily.

Volume of 10 ppm P	Final Volume	Concentration P (ppb)
0.000	50	0
0.125	50	25
0.250	50	50
0.500	50	100
1.000	50	200

Table 1: Dilution scheme for calibration standard preparation

### Procedure

The sample solution should not contain more than 400 ppb of phosphorus present as the orthophosphate and should be neutral. Solutions, which have greater than 400 ppb phosphorus should be diluted or alternatively higher concentration standards can be prepared and used.

Transfer 25 mL of the solution to a 50 mL volumetric flask. Add 5.0 mL of the molybdate solution, followed by 2.0 mL of the hydrazinium sulphate solution. Make to volume with distilled/deionised water and mix well. Immerse the stoppered flasks in boiling water for 10 minutes. Remove the flasks and cool rapidly.

Shake the flasks and measure the absorbance at 830 nm against a reagent blank.

Note that the sample heating for 10 minutes ensures that the reaction has gone to completion. Studies undertaken showed that the blue colour of the phosphorus-molybdenum complex formation was complete after the 10 minute heating and no further reaction occurred. As a stable reading is obtainable immediately after the cooling step, samples can be analysed immediately unlike conventional methods such as the Vanadomolybphosphoric acid method at 470 nm, which has a long stabilization time.

Laboratory productivity is significantly enhanced using the Molybdenum Blue method.

The blue phosphorous-molybdenum complex was found to be stable after 24 hours. This gives the analyst the flexibility that:

1. Samples do not have to be analysed immediately after they are prepared.
2. Sample integrity is maintained if large batches of samples are prepared and then analysed.

immediately after preparation, large sample batches cannot be to be analysed.

## Results

Table 2 lists the standard results obtained. Figure 2 graphically illustrates this data.

Even at the low phosphorus concentrations, a linear correlation was obtained between concentration and absorbance.

Concentration P (ppb)	Absorbance
0.0	0.0000
25	0.0253
50	0.0513
100	0.1042
200	0.2071

Table 2: Calibration results obtained.

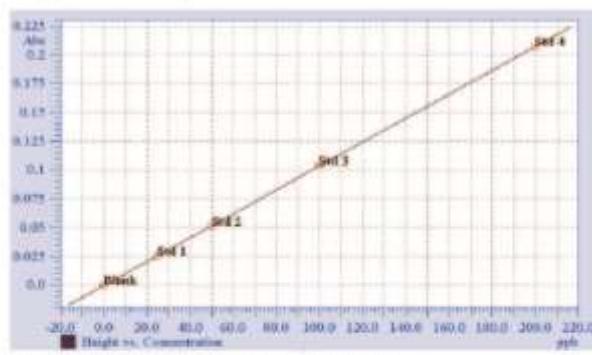


Figure 2: Calibration graph of standards from 25 to 200 ppb Phosphorus.

In contrast, conventional methods such as the Vanadomolybphosphoric acid method at 470 nm do not form stable coloured complexes. Hence with these methods, as samples must be analysed

The detection limit was calculated by analysing a 25 ppb standard and reagent blank 10 times. Using a 3 $\sigma$  confidence limit, a detection limit of 0.2 ppb was obtained. This is 1000 times more.

## **EXPERIMENT E4(I)**

**Introduction to Kinetics: Factors that Affect the Rate of Reaction**

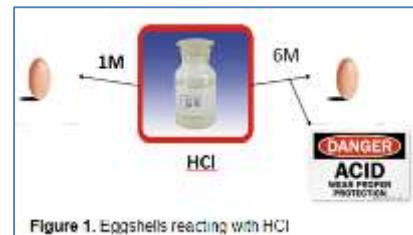
**Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department**

**(Modified version of Chad Eller and University of Michigan General Chemistry**

**Laboratory Manual)**

### **I. OBJECTIVES**

- Be able to list and rationalize the factors that affect the rates of reaction such as that shown in Figure 1.
- Explain various scenarios using the factors that affect reaction kinetics.
- Learn how team works to balance theory with practice



### **II. INTRODUCTION**

Throughout nature, chemical reactions occur at different rates. Some reactions such as the rusting of iron are relatively slow while others such as the combustion of gasoline occur very quickly. Scientists, however, have figured out ways to make various reactions run faster or slower. Becoming familiar with the factors that affect the rate of a reaction gives us insight into how reactions work. The field of chemistry that concerned with the rate at which reaction occurs is called chemical kinetics.

A rough analog can be made to the speed with which a computer completes a specific task. All computers are not created equal. If you try to run the newest 3-D game in high resolution on an old machine you will be lucky to get it to work at all. Each component of a computer has a definite and predictable effect on its performance. Too little RAM, slow bus speed, fragmented hard drive, inefficient operating system or application, multitasking, network congestion – they all work to slow down our computing experience. But for each problem there is a solution. It just takes a little knowledge (theory) and a few tries at improvement (experiments).

Chemists and computer engineers are not the only people concerned with the rates of processes. Consider these examples:

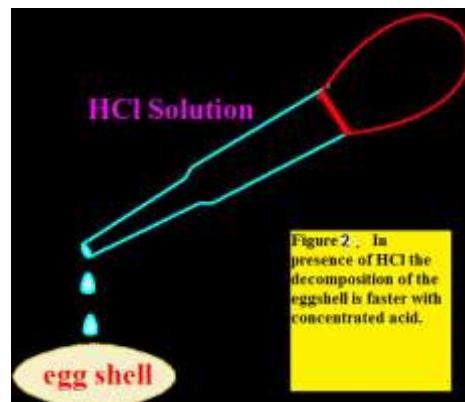
Career	Application
Biologist	Preservation or decomposition of specimens
Chemical Engineer	Speed of production effecting cost
Civil Engineer	Concrete and asphalt curing
Doctor	Medication or poison effecting the body
Museum Curator	Dating, restoration, preservation of artifacts
Restaurant Owner	Food spoilage and safety

## BACKGROUND

In order to understand each factor that affect each the rate of a chemical reaction we can use the simple model of atoms as very small spheres in constant motion. Molecules are groups of these spheres that are bonded together and are constantly bouncing off each other. Picture just a few molecules at a time and consider what happens to them in different circumstances. Think of this model as we study the effects of concentration, surface area (for solids), temperature, and catalysts.

### A. Effect of Changing the Concentration of Reactants

Chemical reactions involve breaking chemical bonds, rearranging the reactant atoms, and making new chemical bonds. In order for this to occur, molecules must collide with each other. If there are only a few molecules of each reactant in a given volume, the number of collision between them will be relatively low. By increasing the concentration of the reactants, we increase the number of reactants molecules in the same amount of space (Figure 2). This means there are more opportunities for a collision to occur.

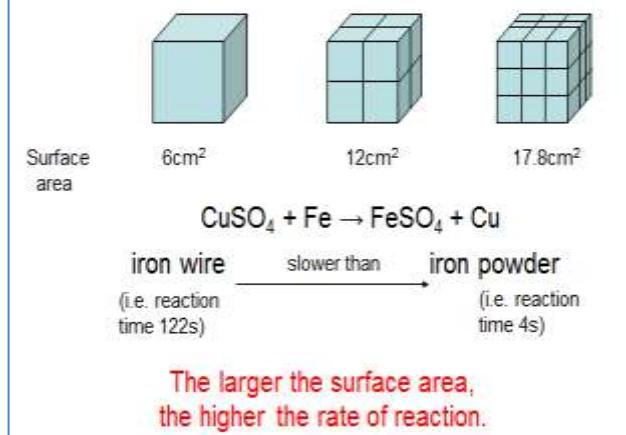


Fishing can be used as an analogue for the effect of concentration. If you are fishing in a well-stocked pond with thousands of fish (a high concentration) you have a better chance of catching a fish than if the same pond only had 2 or 3 fish (a lower concentration).

### B. Effect of Changing the Surface Area

In the case where one of the reactants is a solid, the majority of the atoms are trapped beneath the surface. Only the atoms on the surface are available to collide with the other reactant. When a sample cube is cut into smaller pieces (Figure 3), the amount of surface area increases, even though the volume does not change. Grinding a solid into a powder vastly increases the surface area, making a large portion of the atoms available to collide with the other reactant. In your daily experience, you may have seen that fine salt crystals dissolve in water faster than course rock salt.

Figure 3. Effect of changing cube ( $1\text{cm}^3$ ) surface area



### C. Effects of Changing the Temperature

The average molecular kinetic energy of a sample is constant at a given temperature. However, the random nature of molecular motion means that some molecules will be moving faster than others. At any given temperature a few molecules have enough energy to react. This minimum required energy is called the activation energy,  $E_a$ . As the temperature of the system is increased, the kinetic energy available during collisions goes up and the proportion of collisions exceeding  $E_a$  increases (Figure 4). This allows the reaction to take place faster at a higher temperature.

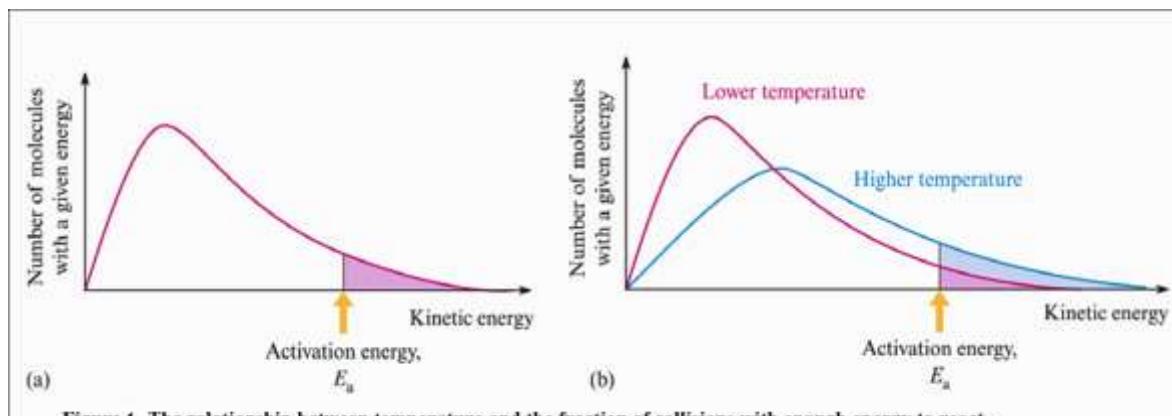


Figure 4 . The relationship between temperature and the fraction of collisions with enough energy to react.

Think of rolling a ball up an inclined driveway into a garage. If you roll the ball slowly, it comes right back to you. When you roll it fast enough, however, the ball makes it into the garage. The amount of the energy needed to get the ball into the garage is analogous to activation energy of a reaction. Only when a molecule can acquire at least that much energy does a reaction take place.

Just as some driveways are steeper than others, chemical reactions differ in the amount of energy needed to make them occur. Why don't all reactions have the same  $E_a$ ? Sometimes the bond to be broken is very strong. In other reactions there is an unstable intermediate molecule that requires a lot of energy to make (Figure 5).

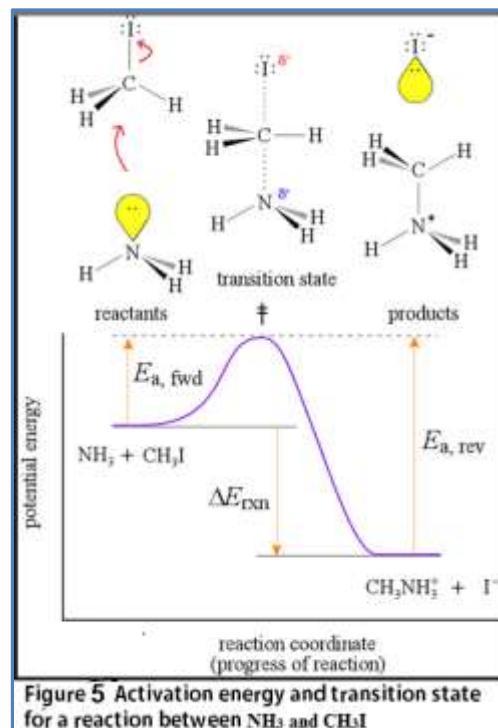


Figure 5 Activation energy and transition state for a reaction between  $\text{NH}_3$  and  $\text{CH}_3\text{I}$

## D. Orientation of the Collisions

As every baseball player knows, hitting the ball does not guarantee a home run. Sometimes the ball hits the top of the bat and pops straight up, other times it hits the bottom of the bat, and the batter grounds out. Only when the swing is aligned with the ball you can hit a home run.

Likewise, in chemistry, every collision does not result in a chemical reaction (Figure 6). In order for bonds to form, atomic orbitals must overlap just right. Complex molecules can have shapes that make it unlikely for this overlapping of orbitals to happen in any particular collision. The likelihood of a correct spatial relationship is expressed in the constant ‘A’, which we will use later in a mathematical model of reaction rates.

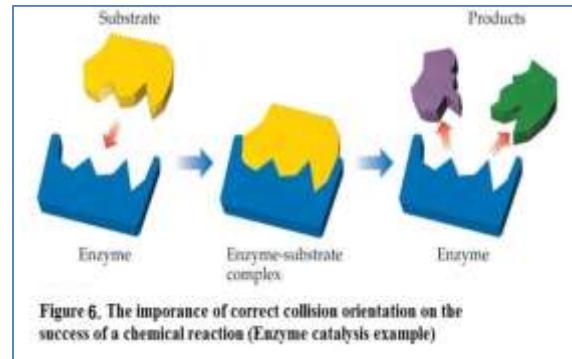


Figure 6. The importance of correct collision orientation on the success of a chemical reaction (Enzyme catalysis example)

## E. Effect of Adding a Catalyst

Although scientists are not able to directly control the activation energy or the orientation of a collision for a reaction, the use of catalyst often allow for the manipulation of these factors. A catalyst is a material that does not permanently change or get used up in a reaction, but helps the reaction run faster. The catalyst lets a reaction from the same product it normally would, but by following a different, less energy intensive route (Figure 7).

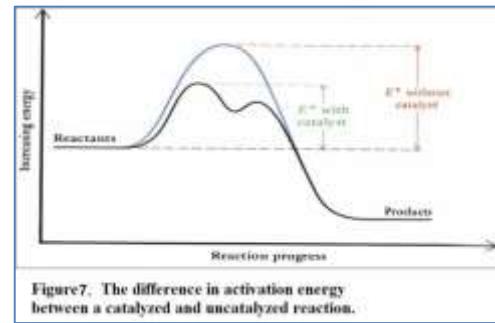


Figure 7. The difference in activation energy between a catalyzed and uncatalyzed reaction.

You can think of a catalyst as bridges: they let you cross the river without having to walk miles and miles to find a shallow spot to wade across. Bridges are used, but not destroyed, and where you end up is the same. They just make the trip easier and faster. Catalysts work in many different and often complicated ways. Sometimes they temporarily donate or absorb electrons, hydrogen ions or hydroxide ions in order to provide the alternative, lower energy reaction mechanism.

## III. OVERVIEW

Each factor that affects the rate of reaction will be demonstrated with a chemical reaction or model. Pay attention to which factor is affecting the rate in each case. A key to understanding kinetics is to consider how the changes we make will determine what the atoms are experiencing thereby leading to a change in the rate of reaction. Before proceeding with the experiment you must follow the safety rules and warning.

Results of experiment E4(I) are mostly qualitative & not quantitative, like observing color change, bubbles etc. So, no need to have precise microgram weights of solid powders of Fe, Zn, MnO<sub>2</sub>, and KI. Save your accurate efforts to complete the next experiment E4(II), so simply compare your sample powder amount to standard sample amounts setting against the wall of the lab. Again, minimize waste and contamination.

**Safety Warning:** Safety rules & chemical waste disposal guidelines must be followed in order to prevent personal injury and to protect yourself, others & the environment. If you are unable to observe the rules then you are at risk of being dismissed from the lab.

**Caution:**

- Do not dump any of the reagents down the sink!
- Discard the waste in an appropriate waste container under the supervision of your instructor!
- Do not allow solutions to come in contact with your skin! Wear gloves & goggles!  
(Silver ion, Ag<sup>+</sup>, will color your skin. Some ions are TOXIC).
- Separate chemical waste of: Acetone, HCl, solutions of (CuSO<sub>4</sub>, I<sub>2</sub>, MnO<sub>2</sub>), H<sub>2</sub>O<sub>2</sub>, & solids to recycle Fe & Zn.
- **Do not waste chemicals and repeat trials not required.**

This is a group experiment where each student shares data with his members of the group, each 2 students in a group must do first experiment E4(I) (Parts A, B, C & E, but once for each part, do not repeat trials). Be conservative on the use of chemicals and do not waste on no need trials. Each student must submit individual report the following week. Do not forget that the individual pre-lab exercises (PLE) due at start of each experiment and should reflect the changes in the procedures shown below.

There are few minor changes to the lab manual instructions of the University of Michigan including skipping minor sections but they are shown by lighted or fading print (shown in case instructor may want you to conduct them).

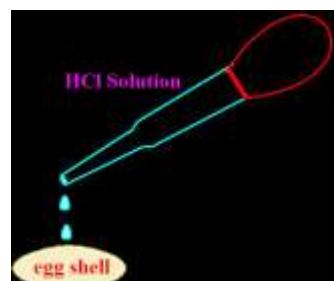
## IV. EXPERIMENTAL PROCEDURES

**“Each 2 students complete the entire experiment E4(I). Take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5”**

### PART A. Effect of Changing the Concentration of Reactants

Chemicals Used	Materials Used
HCl, 1M and 6M in dropper bottles Eggshells	Watch glass (2)

**Caution:** 6M HCl is caustic. If any is spilled on your skin, immediately rinse with running water and inform your laboratory instructor. As always, you should wear your goggles at all times when working in the laboratory. Dispose of waste according to instructor's directions.



1. Bring with you to the lab some eggshells from boiled eggs.
2. Place small pieces of the eggshells in each of the two watch glasses.
3. Add 10 drops of 1M HCl to one sample of eggshells and 6M HCl on the other. Record your observations.

### PART B. Effect of Changing the Surface Area

Chemicals Used	Materials Used
Coffee creamer (Skip this) CuSO <sub>4</sub> , 0.2 M Iron wires & iron powder	Candle (tea light or votive candle) and lighter (Skip this) Spatula Disposable pipet 50 mL beakers (2) Hot plate and thermometer Glass stirring rods

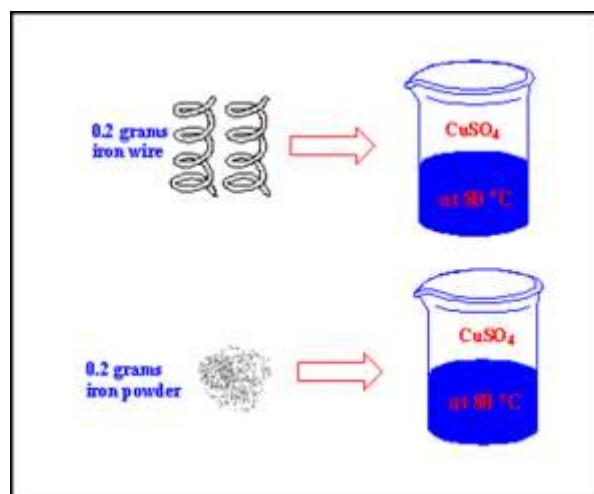
**Caution: Clear flammable materials from lab bench and surrounding areas.**

**Case I:** Flammability of coffee creamer (SKIP THIS CASE I & PROCEED TO CASE II):

After lighting the candle, use a spatula to hold a small amount of coffee creamer in the flame for 5 seconds. Record your observations. Next, draw a small amount of coffee creamer into a disposable pipet. While standing at arms length from the candle, aim a burst of coffee creamer at the flame. Again, record your observations. Clean your lab bench according to your instructor's directions.

**Case II:** Reaction between CuSO<sub>4</sub> and iron metal.

1. Add 5 mL of 0.2 M CuSO<sub>4</sub> to each of two 50-mL beakers and heat both in hot water bath to 80 °C.
2. While solution is heating, prepare separately 1 piece of 0.2g iron wire (0.2g, about 8cm long) and 0.2g iron powder (0.2g, use spatula about 1cm wide of powder from tip). Simultaneously drop the iron samples one into the 1 beakers and the other to the other beaker, then stir.
3. Record any color changes and how long it takes before the changes occur. Dispose the waste according to your instructor's directions.



### PART C. Effect of Changing the Temperature

Chemicals Used	Materials Used
Food coloring (dark) (Skip this)	50-mL Glass beakers (2)
CuSO <sub>4</sub> (0.2 M)	Hot plate and thermometer
Ice	Tongs or heat resistant gloves
Zinc metal (10 mesh, granular)	Spatula, graduated cylinder
	Clay, small lump

#### Case I: Dispersal of dye in hot and cold water (SKIP THIS CASE I & PROCEED TO CASE II).

Fill two 400-mL beakers each with 250 mL water. Heat one to 80 °C. Without stirring, add one drop of food coloring to each container by touching the surface with the dropper. Record the time required for the color to disperse.

#### Case II: Reaction between CuSO<sub>4</sub> and Zinc.

Add in sequence 5 mL portions of 0.2 M CuSO<sub>4</sub> into available graduated cylinder then pour into each of two 50-mL beakers. Heat one solution to 80 °C in the hot water bath, while cooling the other in an ice bath. Add a few pieces of granular zinc (about 0.05g, use spatula about 0.2cm full of powder from tip) to the cold container. Record any color changes and how long it took for the changes to occur. Repeat by adding a few pieces of zinc to the hot solution. Again, record your results. Dispose of waste according to instructor's direction..



#### Case III: Modeling Activation Energy (SKIP THIS PART III).

Roll a piece of modeling clay into a ball no bigger than Ping-Pong ball. Drop the clay on a clean area of floor from a height of 1 foot. Gently push the clay sideways to see if it rolls. Note any shape change upon impact. Drop the clay several more times each time about one foot higher. Roughly how high did you need to drop the clay for it to stick firmly to the floor?

### PART D. Modeling the Significance of the Orientation of Collisions (SKIP THIS PART D & PROCEED TO PART E)

Materials Used
Styrofoam–Velcro balls (4, 2 each with one piece of Velcro and 2 each with 6 pieces of Velcro) Box or deep tray

Place two of the Styrofoam balls with 6 pieces of Velcro on them in a box or deep tray. Gently shake the container until the two balls stick. Repeat with the 2 balls that have only one Velcro square. Describe how readily the balls stick together in each case.

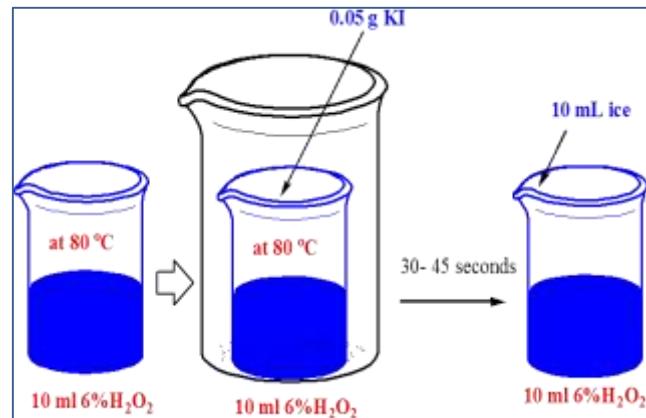
**PART E. Effect of Adding a Catalyst. (Catalyst does not get consumed by the reaction but it donates or absorbs electrons, hydrogen ions or hydroxide ions)**

Chemicals Used	Materials Used
Hydrogen peroxide ( $H_2O_2$ ), 6% solution $MnO_2$ Ice 0.5% corn starch solution (Skip this) Iodine solution (saturated) in dropper bottles	100-mL beaker Hot plate and thermometer Tongs or heat resistance gloves 600-mL beaker Spatula 50-mL beaker Glass stirring rods

**Caution:** Avoid contact with hydrogen peroxide. If any is spilled on your skin, immediately rinse with running water and inform your laboratory instructor. As always, wear your goggles at all times when working in the laboratory. Dispose of peroxide waste according to instructor's directions. In general, peroxide waste is NOT compatible with other chemical waste. Carefully add  $MnO_2$  powder  $H_2O_2$  and avoid direct view of any liquid splashing.

**Case I: Decomposing  $H_2O_2$**

In a 100-mL beaker, heat 10 mL of 6%  $H_2O_2$  to 80°C. Using tongs or heat resistant gloves remove the beaker from the hot plate and place the whole beaker inside a large 600-mL beaker. Using spatula, add roughly 0.05g of  $MnO_2$  (a pinky nail sized granular) to the  $H_2O_2$ . Record your observations within 30-45 seconds. Do not view from above, as minor splashing may occur. After 30 – 45 seconds, add 10 mL of ice to the solution. Again, record your observations.



**Case II: Hydrolysis of Starch. (SKIP THIS CASE II)**

Using 50-mL beakers, warm two 10-mL samples of 0.5% corn starch solution to 40°C. Add 10 drops of iodine to each sample and record your observations. Collect 2 mL of saliva from yourself and lab partner. Thoroughly mix the saliva into one of the corn starch/iodine solutions. Record the time required for the solution to become light lavender or clear. Add a few more drops of iodine. Note the color of drops as they enter solution, and the color after mixing. Dispose of waste according to your instructor's directions.

## **E4(I): Introduction to Kinetics: Factors That Affect the Rate of Reaction**

<b>Name:</b>	<b>Lab instructor:</b>
<b>Date:</b>	<b>Lab section:</b>

### **V. PRE-LABORATORY EXERCISES (PLE)**

1. Name four factors that are under the control of scientist when he or she wants to increase the rate of a reaction.
2. As scientists, we often talk about wanting to speed reactions up. Can you think, however, of any reactions that you might rather slow down than speed up? Can you imagine any way to use the factors from previous question to help you control the specific reaction you want to slow down?
3. You have an important project due tomorrow and you are trying to use every free second to put on the fishing touches. Whenever possible, you also rush through your typical daily tasks and responsibilities. Which of the following has associated with it a time constraint that is under your control and can be adjusted safely: showering, driving to school, math class, eating lunch, Doctor's appointment and grocery shopping. Briefly explain your answers.
4. A living cell must accomplish many complicated chemical tasks. Our bodies contain countless enzymes that are used to speed up otherwise slow reactions. Knowing that enzymes are a type of catalyst, which of the following factors is effected by the presence of an enzyme: the energy of the reactants, the energy of the products, the energy of the transition state, the likelihood of a collision with the correct orientation, or the temperature of the reaction. Briefly explain your answer (s).

## **E4(I): Introduction to Kinetics: Factors That Affect the Rate of Reaction**

Name:	Lab instructor:
Date:	Lab section:

**Important note:** Students of each group must record their experimental raw data on one datasheet of page 13, then copy it (photocopies ok) and include it with this PLQ. In addition, students must complete all the sections on this PLQ.

## **VI. RESULTS AND POST-LABORATORY QUESTIONS (PLQ)**

### **Part A.      Effect of Changing the Concentration of Reactants**

In what ways was the reaction between the eggshells and the 1 M HCl similar to the reaction between the eggshells and the 6 M HCl? In what ways were the reactions different?

### **Part B.      Effect of changing the surface area**

#### **Case I:    Flammability of coffee creamer (SKIP CASE I & PROCEED TO CASE II)**

What was the effect of spraying g the coffee creamer at the flame rather than holding a spatula full in the flame? Explain your observations in terms of surface area.

#### **Case II:    Reaction between CuSO<sub>4</sub> and iron metal.**

Describe the appearance of the solution before addition of the iron metal.

Describe the appearance of the solution and the iron wire after the reaction.

Describe the appearance of the solution and the iron powder after the reaction.

In which case did the reaction occur first? Explain why.

### **Part C.      Effect of Changing the Temperature**

**Case I:** Dispersal of dye in hot and cold water (SKIP THIS CASE I AND PROCEED TO CASE II)

Time required to disperse dye in room temperature water:

Time required to disperse dye in hot water:

Explain your observations in terms of the kinetic energy of the water molecules.

**Case II:** Reaction between CuSO<sub>4</sub> and zinc

Record any color changes & time to occur	<b>Cold solution</b>	<b>Hot solution</b>
<b>Time for 1<sup>st</sup> color change in the....</b>		
<b>Time for 2<sup>nd</sup> color change in the ....</b>		

What effect does increasing the temperature have on the rate of reaction? Explain why.

**Case III:** Modeling Activation Energy (SKIP THIS CASE III)

Why doesn't the clay always stick to the floor? Explain how this activity serves as an analogy for activation energy.

### **Part D.      Modeling the Significance of the Orientation of Collisions (SKIP THIS PART D)**

In which case (the ball with 1 Velcro or 6 Velcro pieces) was the required orientation for successful collision more restrictive? Explain.

Did this agree with your observations of how long it took for the balls to stick? Explain.

## **Part E.           Effect of Adding a Catalyst**

### **Case I:    Decomposing H<sub>2</sub>O<sub>2</sub>**

Observations after addition of KI:

Observations after addition of ice:

In Case I, which material is the catalyst?

Does the temperature affect the usefulness of a catalyst?

### **Case II:    Hydrolysis of Starch (SKIP THIS CASE II)**

Description of corn starch solution:

Observation after addition of Iodine:

Observation after addition of saliva (compare with control):

Observation after addition of more iodine:

1. Marble, like chalk is composed of CaCO<sub>3</sub>. Explain why monitoring the acidity of rainfall would be important with regards to conserving historically and artistically important outdoor statues.
2. A chemical engineer is trying to increase output of a chemical plant. She is considering using an expensive catalyst or increasing the temperature of the large reaction vessel by 20°C to accomplish the same task. Which route will be least expensive in the short term? Long term?
3. Is the blue chemical in the CuSO<sub>4</sub> solution a catalyst? How do you know?
4. As the reaction progresses and the reactants are consumed, will this tend to increase or decrease the rate of reaction? Explain.

## E4(I) DATASHEET FOR A LAB SECTION

*Print this datasheet and bring it with your ALR report, record your raw experimental data on it, then attach it to your individual PLQ.*

VC211 EXPERIMENT E4(I) DATASHEET: KINETICS FACTORS AFFECTING REACTION RATES										
SECTION: _____			TA: _____		EACH 2 STUDENTS COMPLETE ENTIRE E4(I) EXPERIMENT					
PROCEDURE PARTS →			A	A	B	B	C	C	E	
GRP	NAME	ID	CONCENTRATION EFFECT EGGSHELL RXNS			SURFACE AREA EFFECT COLOR CHANGE CuSO <sub>4</sub> @ 80°C 0.2g Fe Wire      0.2g Fe Powder		TEMPERATURE EFFECT COLOR CHANGE CuSO <sub>4</sub> & 0.05g Zn Ice Cold      Hot @ 80°C		ADDING CATALYST EFFECT 10mL 6% H <sub>2</sub> O <sub>2</sub> & 0.05g MnO <sub>2</sub> POWDER 80°C, in 30-40s      10mL ICE AFTER 30-40s
#	Chinese		1M HCl	6M HCl						
8	NOTES →	Add no more than 10 drops	Use available cylinder to measure 5.0 mL and the available hot water bath	Use available cylinder to measure 5.0 mL and the available hot water bath	To avoid back flash avoid direct view and place the sample small beaker inside larger beaker					
9	1									
10	1									
11	1									
12	1									
13	1									
14	2									
15	2									
16	2									
17	2									
18	3									
19	3									
20	3									
21	3									
22	4									
23	4									
24	4									
25	4									
26	5									
27	5									

### IMPORTANT NOTES

- Each two students will do entire E4(I)
- Clean all glassware and rinse with distilled water
- Use 5mL CuSO<sub>4</sub> & hold beaker down with hand while wearing cloth gloves
- Use hot water bath for heating to 80° C
- Handle hot beakers with tong or cloth glove
- Work safely & dispose chemicals in waste container
- Must follow chemical disposal instructions: E4(I) waste in one large beaker (no rinse water), then remove solids into its own waste container, then drain solution into inorganic waste containers, while disposing E4(II) waste into another beaker then remove stirring rod and place on top of stirrer machine pan then dispose the solution in organic waste container

# EXPERIMENT E4(II)

## Determining the Rate Law: A Kinetics Study of Iodination of Acetone

Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department

(Modified version of Kristen Spotz and University of Michigan General Chemistry Laboratory Manual)

## I. OBJECTIVES

- Gain a quantitative understanding of kinetics.
- Determine the rate of a reaction, the order of the reaction with respect to the reactants and the value of the rate constant.
- Predict reaction times using an experimentally determined rate law.

## II. INTRODUCTION

One factor influencing the rate of the reaction is the concentration of the reactants. Typically, as the concentration of the reactants increases so does the rate of the reaction. The actual relationship, however, can be quite complicated. Sometimes, doubling the concentration of a reactant will result in a doubling of the rate. For other chemical reactions doubling the concentration of a reactant might have no effect on the rate or it might result in a four – fold increase in the rate. It can be useful for the scientist to have understanding of the relationship between the concentration of the reactants and the rate of the reaction. For example, for a quantitative knowledge of reaction rates, scientists are able to gain insight into reaction mechanisms and even predict the time frame of a reaction. Detailed study by 1995 Nobel laureates F. Sherwood Rowland and Mario Molina of the rates of hundreds of chemical reactions provided insight into the role of chlorofluorocarbons (CFCs) in the depletion of the ozone layer (Figure 1). So, how do scientists determine the relationship between concentration and reaction rate and how are the results expressed in a useful form?

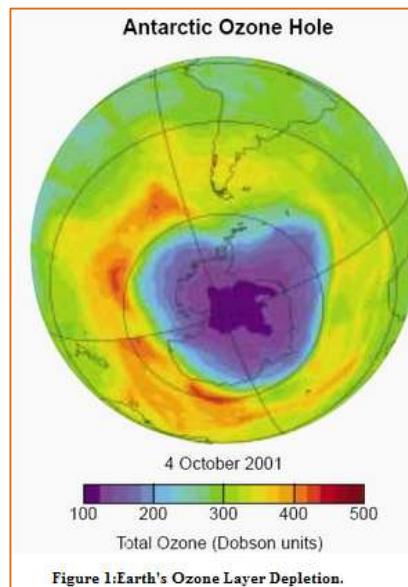
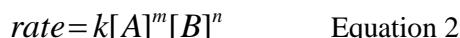


Figure 1: Earth's Ozone Layer Depletion.

## III. BACKGROUND

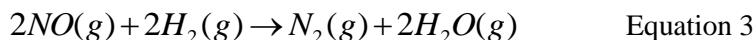
### A. The Rate Law

All the information needed to predict the rate of a given reaction is contained in the rate law, or the rate equation. Given the generic reaction in equation 1, the general form of the rate law would be given in Equation 2:



The rate constant,  $k$ , is specified for each reaction and is temperature dependent. The units for the  $k$  are dependent on the overall order of the reaction. The rate law also includes the concentrations of the reactants raised to the reaction orders,  $m$  and  $n$ . The values of  $m$  and  $n$  must be determined experimentally (as you will do in today's experiment) and cannot be derived from the balanced chemical equation.

As an example, we will examine the reaction (equation 3) and the experimental data (table 1) for the reaction of nitrogen monoxide and hydrogen gas to produce nitrogen gas and steam.



Exp #	[NO] <sub>initial</sub> M	[H <sub>2</sub> ] <sub>initial</sub> M	Initial rate M/s
1	0.1	0.1	1.23×10 <sup>-3</sup>
2	0.1	0.2	2.46×10 <sup>-3</sup>
3	0.2	0.1	4.92×10 <sup>-3</sup>

The first step in determining the rate law is to follow the example in equation 2 and write the general form of the rate law for the reaction (Equation 4)

$$Rate = k[NO]^m[H_2]^n \quad \text{Equation 4}$$

The next step is to find the order for each reactant. In order to find the order with respect to hydrogen gas, experiment #1 and #2 are compared

$$\frac{Rate_2}{Rate_1} = \frac{2.46 \times 10^{-3}}{1.23 \times 10^{-3}} = \frac{k[0.10]^m[0.2]^n}{k[0.10]^m[0.1]^n} \quad \text{Equation 5}$$

The rate constants, k, and the concentrations of nitrite will cancel (along with the unknown m) leaving equation 6:

$$2 = 2^n \quad \text{Equation 6}$$

Mathematically, n=1. In terms of kinetics, this is interpreted to mean that the reaction is first order with respect to hydrogen gas. This result makes sense if we look back at table 1. Comparing experiment 1 and 2, if the concentration of nitrogen monoxide is held constant and we double the concentration of hydrogen, the rate of the reaction doubles.

The same process (Equation 5) is taken to determine the order with respect to nitrogen monoxide. From setting up the ratio of experiments 1 and 3, the order with respect to nitrogen monoxide is determined to be second order. This means that if the concentration of hydrogen is held constant while doubling the concentration of nitrogen monoxide, the rate of the overall reaction quadruples. The overall order of the reaction (n+m) is 3.

Now that we know the order of the reaction, the next step is to know the value of the rate constant, k. the units for the k are dependent on the overall order of the reaction. Data from any experiment given in the table may be used to determine the rate constant. For example, using the data from experiment #1 results in Equation 7.

$$1.23 \times 10^{-3} \text{ M/s} = k[0.10 \text{ M}][0.1 \text{ M}]^2 \quad \text{Equation 7}$$

Solving for the rate constant, the value is equal to 1.23M<sup>-2</sup>s<sup>-1</sup>. The final rate law includes the value for both the rate constant and the orders of the reaction.

$$Rate = 1.23 \text{ M}^{-2} \text{ s}^{-1} [H_2][NO]^2 \quad \text{Equation 8}$$

Using equation 8 above, any initial concentration of hydrogen gas and nitrogen monoxide can be inserted into the rate law in order to predict the rate of the reaction.

In another similar example, the method involves a series of experiments in which the initial concentrations of some reactants are held constant and others are varied in convenient multiples in order to determine the rate law for that reaction. The following is the data for the gaseous reaction: 2NO(g) + Cl<sub>2</sub>(g) = 2NOCl(g)

Experiment	Initial [NO]	Initial [Cl <sub>2</sub> ]	Initial Rate, M s <sup>-1</sup>
1	0.0125 M	0.0255 M	2.27 × 10 <sup>-5</sup>
2	0.0125 M	0.0510 M	4.55 × 10 <sup>-5</sup>
3	0.0250 M	0.0255 M	9.08 × 10 <sup>-5</sup>

Show that the following rate expression applies: Rate =  $k [NO]^2 [Cl_2]$

In general, set Rate =  $k [NO]^m [Cl_2]^n$  use data above to find m, n, & k.

$$2.27 \times 10^{-5} = k (0.0125)^m (0.0255)^n \quad \dots \dots \dots \text{Eq.1}$$

$$4.55 \times 10^{-5} = k (0.0125)^m (0.0510)^n \quad \dots \dots \dots \text{Eq.2}$$

$$9.08 \times 10^{-5} = k (0.025)^m (0.0255)^n \quad \dots \dots \dots \text{Eq.3}$$

$$\text{Eq.2/Eq.1: } 2 = 2^n, \text{ then } n = 1$$

$$\text{Eq.3/Eq.1: } 4 = 2^m, \text{ then } m = 2$$

The overall order of reaction =  $m + n = 3$ , first order with respect to  $Cl_2$  gas & second order with respect to NO gas.

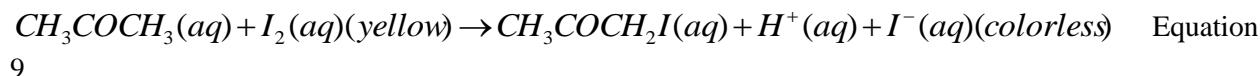
Using any of the equations, then k is solved:

$$k = 5.70 \text{ M}^{-2} \text{ s}^{-1}$$

$$\text{Rate} = 5.70 \text{ M}^{-2} \text{ s}^{-1} [NO]^2 [Cl_2]$$

## B. The Iodination of Acetone

In today's experiment, you will be studying the kinetics of the reaction between acetone and iodine to form iodoacetone and iodide (Equation 9).



The rate law will be determined by varying the concentration of acetone and iodine. To study reaction rates it is necessary to measure the concentration of reactants as a function of time at the start of the reaction (the "initial rate" in table 1), making kinetics studies typically difficult. In this particular experiment, however, the amount of acetone will be kept in vast excess with respect to the amount of iodine so that the concentration of acetone does not change appreciably during the course of the reaction. As a result, the rate of the reaction remains relatively constant throughout the course of the reaction. In other words, the "initial rate" that we need in order to determine the rate law will be equated with the average rate of the reaction. The equation used to find the average rate of reaction (equation 10) is found by measuring the change in iodine concentration divided by the time needed to react.

$$\text{Rate} = -\frac{-\Delta[I_2]}{\Delta t} = \frac{-(I_2)_{\text{final}} - (I_2)_{\text{initial}}}{\Delta t} \quad \text{Equation 10}$$

The study of the reaction for the iodination of acetone is also made easy due to the color ranges of the solution. Iodine ( $I_2$ ) is a pale yellow whereas the iodide ion ( $I^-$ ) is colorless.---acid, which is introduced to the reactant solution as a catalyst, is also colorless. Therefore, changes in iodine concentration can easily be visualized. The time at which the pale yellow color of the initial solution turns clear indicates that the reaction is completed and that  $[I_2]_{\text{initial}} = 0 \text{ M}$ .

## IV OVERVIEW

Follow the overview carefully and summarize in 1-2 sentences in your report.

Results of Experiment E4(II) are mostly quantitative so you need to be precise in conducting the experiment and collecting the data. This is a group experiment but you must submit individual report

along with the ALR of Experiment E4(I). Do not forget that the individual pre-lab exercises (PLE) due at start of this experiment.

**Safety Warning:** Safety rules & chemical waste disposal guidelines must be followed in order to prevent personal injury and to protect yourself, others & the environment. If you are unable to observe the rules then you are at risk of being dismissed from the lab.

**Caution:**

- Do not dump any of the reagents down the sink!
- Discard the waste in an appropriate waste container under the supervision of your instructor!
- Do not allow solutions to come in contact with your skin! Wear gloves & goggles!  
(Silver ion,  $\text{Ag}^+$ , will color your skin. Some ions are TOXIC).
- Separate chemical waste of: Acetone, HCl, solutions of ( $\text{CuSO}_4$ ,  $\text{I}_2$ ,  $\text{MnO}_2$ ),  $\text{H}_2\text{O}_2$ , & solids to recycle Fe & Zn.
- [Do not waste chemicals and repeat trials not required.](#)

--- following a procedure for the preparation of solution 1, you will devise your own experimental protocol for creating solution 2,3 and 4 in order to determine the order with respect to acetone and iodine, the value of the rate constant, and predictions concerning reaction rate. Remember for Step 6 in the procedure decide with your partner by proposing to the TA how to change the concentrations of each of the reactants by varying the volume of each in a manner to yield easily the values of the reaction orders m & n, as illustrated by the example in the manual for this experiment. Have your TA approve the proposal before proceeding. The uncompleted data table shown in this procedure is a guideline table that you may use to propose your work for this part of the experiment and then complete the table. **The data table at the end of the procedure must be completed as part of the PLE & PLQ, but ahead of the lab session. Then your TA inspects the table and if approved you can proceed with this experiment.**

## V. EXPERIMENTAL PROCEDURE

“Students work in pairs / Copy & complete the data table in this section under Calculations & Data Recording, as part of your PLE & also PLQ (show calculations & record the data in the table before starting the experiment). Each student must complete one design reaction.

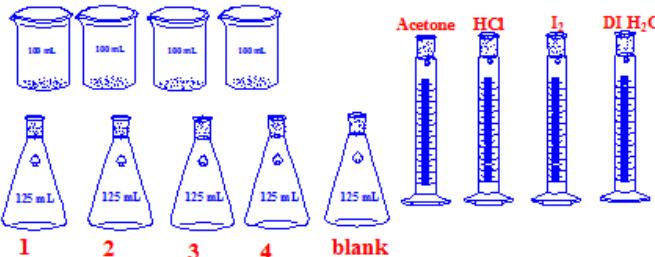
Chemicals used	Materials used
4 M Acetone (100 mL)	50 mL graduated cylinders (4)
1M HCl (100 mL)	100 mL Beakers (4)
0.00118 M Iodine (100 mL)	125 mL Erlenmeyer flasks (5)
De-ionized water (DI $\text{H}_2\text{O}$ )	Plastic pipet (4) Stop – watch Stir – plate and stir bar (optional)

**“Make sure you take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5”**

- Thoroughly clean four 100-mL beakers, four 50-mL graduated cylinders [one cylinder for each acetone (reactant B), HCl solution (acts as catalyst only), I<sub>2</sub> (iodine reactant A), & de-ionized water (for dilution)], and five 125-mL Erlenmeyer flasks (for 4 samples & 1 blank) with soap and water. Rinse all the glassware with deionized water and allow drying. Label one of each beaker and graduated cylinder with the following: "Acetone", "HCl", "I<sub>2</sub>", "DI H<sub>2</sub>O". Label four of Erlenmeyer flasks with number 1 – 4 and label the flask "blank" (see figure below). Rinse each beaker/graduated cylinder with 2 – 3 mLs of the solution named on the label. Pour approximately 50 mL of the appropriate solution into the labeled 100-mL beakers.

### Clean the glassware.

**soap solution → tap water → de-ionized water**



- Prepare a blank that you will use for a color comparison. The blank should consist of 50 mL of water in a 125-mL Erlenmeyer flask. The changes in the clarity of the solution colors during reactions for samples 1 – 4 will be compared to the clarity of this blank DI H<sub>2</sub>O sample. To view colors clearly, place each flask during the reactions over white paper.



- With the appropriately labeled 50 mL graduated cylinders, add 10.0 mL of acetone, 10.0 mL of HCl and 20.0 mL of deionized water into a clean 125 mL Erlenmeyer flask (labeled solution #1).



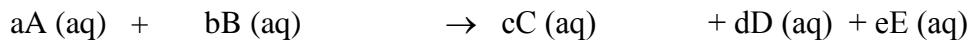
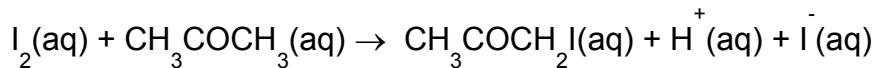
- Get your stop watch ready, measure 10.0 mL of 0.00118 M iodine into the clean "I<sub>2</sub>" graduated cylinder. Place the Erlenmeyer flask (solution #1) onto the stir – plate and drop carefully a stir bar into the solution. Set a stir plate to a medium setting. Get the stop watch started as soon as you quickly pour all the iodine solution into the Erlenmeyer flask. Immediately begin timing the reaction as soon as all the iodine has been transferred to solution #1. The solution will appear yellow due of iodine. The color will fade as the iodine react with the Record the time when the color of iodine just disappears by with the blank. Record the volumes of acetone, HCl, iodine and H<sub>2</sub>O for solution #1 in the data table at the end of this section. Do not use the heating mode on the stir plate (do not heat sample).
- Repeat steps 3 and 4 only once, using same amount of chemicals. Calculate the percent difference (of times to complete the reactions) between the trials. When you careful, the percent difference is less than 5%. Unfortunately, to reduce chemical waste you cannot do more than 2 trials total for each of the 4 samples, so work diligently.

to the presence  
acetone.  
comparison



6. With your partner, decide how to alter the composition of solution #1 to determine the order with respect to iodine (solution #2) and the order with respect to acetone (solution #3). The only requirement is that you must maintain a total volume of 50 mL and the volume of HCl must be 10 mL. After showing your proposal to your instructor, carry out the reactions for solutions #2 and #3 exactly as described for solution #1. If you are careful then no need to repeat trials to 95% accuracy.
7. For your final reaction, devise solution #4 using reactant volumes that you have not previously used. Remember, the total volume must remain 50 mL and that 10 mL of the total volume must be 1 M HCl. Record the time of the reaction.
8. Summarize all your data by completing the attached table in the next section. Using the data to determine the reaction rate order (m) with respect to  $I_2$ , the reaction rate order (n) with respect to acetone, and the rate constant (k). Then write the final equation for the rate expression.

**Calculations & Data Recording:** Every group/student must complete data below as part of the pre-lab & before the start of the lab session. Then show that to your ta's for his approval before proceeding with the experiment. Include this section in both PLE & PLQ.



$$R_A = -\Delta[I_2] / \Delta t = -([I_2]_{\text{final}} - [I_2]_{\text{initial}}) / (t_2 - t_1)$$

Or in general:  $R_A = k[A]^m[B]^n$

where [A] & [B] are initial conc. of  $I_2$  & acetone consecutively at time -0- seconds.

$$R_A = k[A]^m[B]^n \dots \text{Eq.1, but in dilution } C_{\text{conc}} \cdot V_{\text{conc}} = C_{\text{dil}} \cdot V_{\text{dil}} \dots \text{Eq.2}$$

$$\text{or } C_{\text{dil}} = C_{\text{conc}} \cdot V_{\text{conc}} / V_{\text{dil}} \dots \text{Eq.2}$$

$$\text{but } R_{A1} = k_1 [C_{A1}/V_{A1}]^m [C_{B1}/V_{B1}]^n \text{ or}$$

$$R_{A2} / R_{A1} = (k_2/k_1) [C_{A2}/V_{A2}]^m [C_{B2}/V_{B2}]^n / ([C_{A1}/V_1]^m [C_{B1}/V_{B1}]^n) \dots \text{Eq.3}$$

Using Eq.2 into Eq.3 and applying the equations above at constant temperature ( $k=k_1=k_2$ ) to data of Sample #1, #2 & 3, to get the reaction order m, n & the reaction rate constant k.

Remember  $V_1 = V_2 = V_3 = V_4 = V_{\text{dil}} = 50 \text{ mL}$ , while  $V_{\text{conc}}$  is the proposed design volumes  $X_i$  or  $Z_i$  in the table below so the distilled water volume is  $Y_i = 50 - X_i - Z_i - 10 \text{ mL}$ , and the starting concentrations  $C_{\text{conc}}$  are given below for each solution.

**DATA TABLE: Proposed lab work to determine the reaction rate orders m & n and the reaction rate constant k.**

SAMPLE #	4M Acetone mL	H <sub>2</sub> O mL	1M HCl mL	0.00118 M I <sub>2</sub> mL	Total Vol. mL	Initial M Acetone in 50mL Moles/L	Initial M I <sub>2</sub> in 50mL Moles/L	Trial 1 Rxn time s	Trial2 Rxn time s	Avg. Rxn time s
1	10.0	20.0	10.0	10.0	50.0	?	?	?	?	?
2	X1	Y1	10.0	Z1	50.0	?	?	?	?	?
3	X2	Y2	10.0	Z2	50.0	?	?	?	?	?
4	X3	Y3	10.0	Z3	50.0	?	?	?	?	?
BLANK	0	50.0	0	0	50.0	Compare sample solution color to water sample transparent color				

**Notes:**

- The total volume should be 50.0mL, so Y values should be the difference between 50.0mL and what you proposed for the other solutions.
- Initial concentrations are shown at time 0s inside the 50.0mL solution and not the starting concentrated stock solution. Use  $V_1x C_1 = V_2x C_2$  to get  $C_2$
- The initial concentration. HCl acts as a catalyst and will not appear in the reaction rate expression.
- Sample #4 is to be used for the last part of the Step 7.
- At the end do not forget to write into the **PLQ** the complete reaction rate with values of m, n & k in the rate expression. It is OK to copy and complete the above table into **PLE or PLQ** to get the instructor's approval prior to starting the experiment, then enter the results on the same table after completing the experiment.
- When experiment is complete, remove the stir bar from the reaction flask using the metal side of a wire brush and do not discard the stir bar in waste containers or sink. If you do then you will remain in lab until you recover the stir bar and give to instructor or you will receive no credit for the experiment.

**ALSO, RECORD YOUR RESULTS ON THE DATASHEET FOR THIS EXPERIMENT**

**E4(II): Determining the rate law: a kinetics study of the iodination of acetone**

Name:	Lab instructor:
Date:	Lab section:

**VI. PRE-LABORATORY EXERCISES (PLE)**

1. According to your text book, what are the four factors that affect the rate of a chemical reaction? Which of these factors will be studied in this experiment?
  2. Distinguish among the following terms: initial rate, average rate, and instantaneous rate.  
Which of these rates would you expect to have the largest value? Explain.  
Which of the rates are typically used to determine the rate law for a reaction?  
Which of these rates will we use to determine the rate law for a reaction?
  3. Use equation 9 to predict the initial rate if  $[NO]_{initial}=0.30\text{ M}$  and  $[H_2]_{initial}=0.15\text{ M}$ .  
What would happen to the initial rate of the reaction if  $[NO]_{initial}=0.60\text{ M}$  and  $[H_2]_{initial}=0.15\text{ M}$  instead. Does your result make sense in terms of the order of the reaction?
  4. Assuming that concentrations are expressed in moles per liter and time in seconds, what are the units of the rate constant, k, for an overall first order rate law? Show your work.  
What are the units of k for an overall second order rate law? Show your work
- Using these two rate constants as examples, write a general rule to explain how the units of the rate constant depend on the overall order of the rate law.
5. Write the general form of the rate law for the reaction in Equation 9.

**E4(II): Determining the rate law: a kinetics study of the iodination of acetone**

Name:	Lab instructor:
Date:	Lab section:

**VII. RESULTS AND POST-LABORATORY QUESTIONS (PLQ)**

“Use the following tables or the table at the end of the procedure to record your calculations and data.” Also, the students of each group must record their experimental raw data on one datasheet of page 11, then copy it (photocopies ok) and include it with this PLQ. In addition, students must complete all the sections on this PLQ.

	Solution #1	Solution #2	Solution #3	Solution #4
Volume, 4.0 M acetone				
Volume, 1 M HCl				
Volume, 0.00118 M iodine				
Reaction time, trial 1				
Reaction time, trial 2				
Average reaction time				

For solution #1, only record the times for the two trials within 5%.

Summarize your results as in table 1 in the background:

Solution	[Acetone] <sub>initial</sub> (M)	[iodine] <sub>initial</sub> (M)	Initial rate (M/s)
1			
2			
3			
4			

“be sure to account for your dilution to 50 mL.

“use Equation 10 in Background.

1. Determine the rate law (including the values for the orders of the reaction and the value for the rate constant with units) for the reaction studied in this experiment. Show all your work.

2. Use the rate law to make a prediction for theoretical initial rate of the reaction for solution #4. How does it compare to the experimental initial rate for solution #4?
  3. Why is it important to keep the total volume of solutions #1-4 at 50 mLs? If more water had been introduced to one of the solutions (giving a total volume of 60 mLs), would you expect the reaction rate to increase or decrease? Explain.
4. The following reaction occurs without a change in the color.  $2A(g) + B_2(g) \rightarrow 2AB(g)$
- a. How could you monitor the concentration of the reactants and products?
  - b. How would you determine the reaction orders?
  - c. How would you find the rate constant and the units for the rate constants?

## SAMPLE DATASHEET FOR A LAB SECTION

*Print this datasheet and bring it with your ALR report, record your raw experimental data on it, then attach it to your individual PLQ.*

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P			
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16			
<b>VC211 EXPERIMENT E4(II) DATASHEET: DETERMINING THE RATE LAW</b>																		
TA: _____				LAB ROOM: _____														
$I_2(aq) + CH_3COCH_3(aq) \rightarrow CH_3COCH_2I(aq) + H^+(aq) + I^-(aq)$																		
$aA + bB \rightarrow cC + dD + eE$				$Rate = k [A]^m[B]^n$														
Procedure Part ----->				GROUP EXPERIMENT BUT EACH STUDENT MUST DESIGN, PREPARE & TEST ONE SAMPLE. SUBMIT INDIVIDUAL REPORTS												<b>RATE CALCULATIONS</b>		
7	8	9	10	SAMPLE	1M HCl ml	4M Acetone X mL	0.00118M D-I Water Y mL	INT. M Iodine Z mL	INT. M Acetone Moles/L	INT. M Iodine Moles/L	TRI. 1 RXN. Time t1 (s)	TRI. 2 RXN. Time t2 (s)	AVG. RXN. Time t <sub>avg</sub> (s)	m	n			
11	12	13	14	#	Chinese	ID												
15	16	17	18	EXAMPLE:	10	10	20	10										
19	20	21	22	1	1	2	3	4										
23	24	25	26	2	2	3	4	1										
27	28	29	30	3	3	4	1	2										
31	32	33	34	4	4	1	2	3										
35	36	37	38	5	5	1	2	3										
39	40	41	42	5	5	2	3	4										
43	44	45	46	6	6	3	4	1										
47	48	49	50	-	-	-	-	-										

### IMPORTANT NOTES:

- Each student must do one reaction of entire E4(II)
- Clean all glassware and rinse with distilled water
- Use graduated cylinders to measure volumes directly

**From stock solutions, do not use beakers**

- Must stir reaction without heating & splashing starting low setting
- Compare reaction color change to blank water sample – glassware over white paper
- Must not dispose stirrer rod in sink or waste container

**You will be penalized heavily if you do not follow**

- Work safely & dispose chemicals in waste container
- Must follow chemical disposal instructions: E4(I) waste in one large beaker (no rinse water), then remove solids into its own waste container, then drain solution into inorganic waste container, while E4(II) waste in another beaker then remove stirring rod and place on top of stirrer machine pan then dispose the solution in organic waste container

**EXPERIMENT E5**  
**Precipitation and Water Purity**  
**Prof. T. Hamade, UM-SJTU JI & SJTU Chemistry Department**  
**(Most photos VC211 previous labs/Hamade)**

**(Modified version of University of Michigan General Chemistry Laboratory Manual<sup>1</sup>)**

**I. AFTER-LAB REPORT (ALR) INSTRUCTIONS FOR EXPERIMENT E5**



This is a group experiment but each student must submit the entire individual report by the end of the experiment E5, however data analysis and discussions can be shared among the group members. You must adhere to all safety rules.

**So prepare all the following report sections entirely ahead of time. At end of experiment you must collect all below sections and give to instructor before leaving the lab.**

1. Type cover page (format same as instructed before).
2. Study ahead of lab (using references, internet and library resources) section IV. PRE-LAB ASSIGNMENTS as instructed and follow section V. GENERAL INSTRUCTIONS.
3. Copy or type from this document and from references a brief description (no more than 1 page total) of (sections II, III & references at end of this document): objectives, introduction, background, and theory. To help you with this, you may use your own typed summary of the quoted references and the additional references at the end of the report (do not include the additional references in your report). Again no more than one page for this section.
4. Ahead of time, read & follow instructions of experimental procedures in sections VI.
5. Copy/paste/type the procedures given in section VI (your choice to copy as is, no need to handwrite). Leave some spaces as needed to handwrite your data and notes.
6. There are no PLE or PLQ for this experiment, however, you must copy/paste/type (as is the entire section VII. REPORTS OF RESULTS) to include the following portions that appear before the additional references at the end of this document. **Leave enough space to handwrite yours/team answers during and immediately after the experiment is completed: (ignore all faded text sections that are identified as “skip this part”)**
  - a. Pre-Laboratory Report: Answer the questions ahead of lab.
  - b. Team Report: All parts 1 through 5
  - c. Team Assessment Form
  - d. Laboratory Discussion Team / Presentation Grading Form
  - e. Laboratory Discussion
  - f. Grading (must include in report for instructor to complete or you get no grade)



## **SUMMARY OF E5 ALR REPORT (include cover page)**

- I. AFTER-LAB REPORT (ALR) INSTRUCTIONS FOR EXPERIMENT E5**
- II. OBJECTIVES**
- III. INTRODUCTION & BACKGROUND**
- IV. PRE-LAB ASSIGNMENT**
- V. GENERAL INSTRUCTIONS**
- VI. EXPERIMENTAL PROCEDURES (ignore all faded text sections that are identified as “skip this part”)**

### **PART 1. *What is a Precipitate?***

- a. Information
- b. Procedure

### **PART 2.A. Is Precipitation Predictable**

- a. Information
- b. Notes to the Procedure
- c. Procedure
- d. Data Analysis
- e. Optional Points to Consider (**skip this part**)

### **PART 2.B. *Can I Identify it?* (**skip this part**)**

### **PART 3. *Concentration and Precipitation***

- a. Information
- b. Notes to the Procedure
- c. Procedure
- d. Additional Information
- e. E. Data Analysis: Use Table 3, Table 4 & Table 5 to record your results
- f. Extensions (**skip this part**)
- g. Optional Points to Consider (**skip this part**)

### **PART 4. *Solvent Pollution & Precipitation***

- a. Information
- b. Notes to the Procedure
- c. Procedure
- d. Data Analysis
- e. Optional Points to Consider (**skip this part**)

### **PART 5. *Can I purify it?* (**skip this part**)**

## **VII. REPORTS OF RESULTS**

Pre-Laboratory Report

Team Report: Parts 1-5

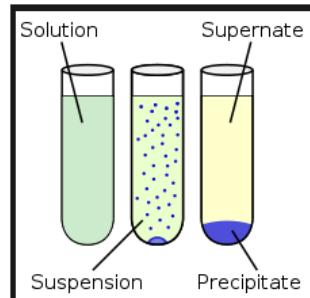
Team Assessment Form: Instructor may have different evaluation form

Grading (**skip this page**)

## **REFERENCES**

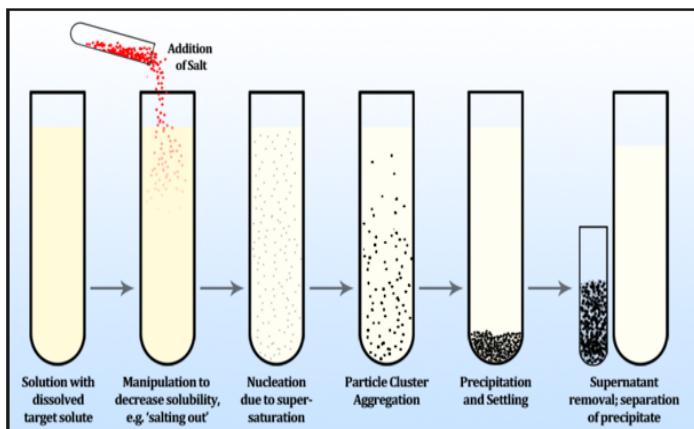
## II. OBJECTIVES

- Become familiar with the logic and experimental tests used to determine precipitate identity.
- Investigate links between precipitation and ion characteristics.
- Determine if some minimum ion concentration is required for precipitation.
- Compare the impact of water and solvents other than water on water purity and precipitation.
- Design experiments to identify unknown ions in a sample of water.



## III. INTRODUCTION & BACKGROUND

Waterborne chemicals pose a threat to the safety and purity of water supplies. Soluble forms of many of the heavy metals (such as copper, mercury, and lead ions) are toxic. Soluble forms of the alkaline earth metals (such as calcium and magnesium ions) cause water hardness of the most common manifestations of water hardness is the insoluble scum formed in the presence of soap. Soluble calcium ions in the water and stearate ions in soap can react to form insoluble calcium stearate. The solid formed when the dissolved ions come together is the "precipitate". The dumping of liquids other than water may cause additional insoluble scum to form.



Your task is to determine by experiment the answer to questions about water purity and precipitation. For example, is precipitation a predictable event? You will also design experiments to identify or remove undesirable contaminants (ions) from water by precipitation.

## IV. PRE-LAB ASSIGNMENT

Study the following references and some of the references duplicated at the end of this document for E5:

1. University of Michigan laboratory manual: Konigsberg Kerner & Penner-Hahn, Hayden McNeil; Hands on Chemistry Laboratory Manual, 1st Ed., Jeffrey A. Paradis, Kristen Spotz, McGraw Hill Higher Education Press, 2006 (Experiment 6- Precipitation and water Purity pages 49-73, Appendix C.1 Precipitation and Solubility pages 185-188, and Appendix A.3 Filtration page 167 ).
2. VC210 textbook: Chemistry-The Central Science (Theodore L. Brown; H. Eugene LeMay, Jr.; Bruce E.Bursten), Pearson International edition (Pearson Prentice-Hall, Inc.), 12<sup>th</sup> edition 2012, ISBN 9780321696724 (Chapter 15 Tab le 4.1 page 121 and Chapter 17 pages 722-739). Topics:

solubility guidelines for common ionic compounds in water, solubility equilibria, factors that affect solubility, precipitation & separation of ions, & qualitative analysis of metallic elements.

## V. GENERAL INSTRUCTIONS

**"Make sure you take photos of your favorite lab work for use in your final PPT presentation assigned by your TA about one of the experiments E1-E5. TA will examine your data immediately after the experiment and no need to record your data on his/her typical datasheet"**



This is a group experiment where each student shares data with his members of the group & groups share data with each other as instructed below for each part of the experiment. At the end of the experiment you must turn in individual reports immediately before leaving the laboratory or you will get -0- points for this experiment.

- 1. You should follow safety rules, regulations and the guidelines on chemical waste disposals. Protect yourself, your neighbor & the environment.**
- 2. At start of lab after my lecture, TA will walk around the groups, collect E4 reports, examine the pre-lab reports of E5 as directed in section I, and then each group give brief few minutes discussion of last week experiment.**
- 3. Immediately as soon as you finish your experiment, each group will gather one after the other in front of the TA and discuss your results and conclusions of today's experiment.**
- 4. After that, you must start cleaning your entire work bench areas, including disposing waste in proper containers and wash glassware with soap & water using brush.**
- 5. Then after that the instructor will inspect your cleaned area and if he satisfied he will tell you to proceed discussing the results with your teams and prepare the final team report so you can give that to the TA before leaving the lab.**



## VI. EXPERIMENTAL PROCEDURES

**Record all the raw data on the datasheet of the page 17**

Copy/paste/type your own merged version of experimental procedures given in the following entire section. Section VI has instructions on which procedures to follow or skip. Leave some spaces as needed to handwrite your data and notes. To eliminate confusions just follow procedure in section VI instead of previously used University of Michigan laboratory manual.

### PART 1. What is a Precipitate?

Your goal is to design experiments and use your logic to determine precipitate identity.

#### a. Information (see reference Chapter 4, Table 4.1, page 121 of VC210 textbook)

Potassium ( $K^+$ ), sodium ( $Na^+$ ) and nitrate ( $NO_3^-$ ) ions are highly soluble (see references).

## b. Procedure

Each 2 students in a group must test 1 sample once, so each group will test 2 samples (once each).

### CAUTION

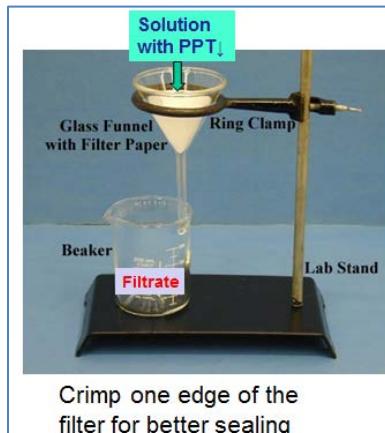
Do not dump any of the reagents down the sink.

Discard the waste in an appropriate waste container.

Do not allow the solutions to come in contact with your skin.

1. Obtain 5ml of 0.1M copper sulfate, CuSO<sub>4</sub>, and 5mL of 0.1M barium chloride, BaCl<sub>2</sub>. Record the appearance of each solution.
2. Combine each solution into one small beaker. Record your observations. Label and save the mixture for later use.
3. Assuming that the reaction involves the coming together of ions in solution, what ions could have combined to form the precipitate?
4. Separate the reaction mixture by filtration (see demo figures on the right and also see end of experiment). Record the observable properties of the filtrate. What conclusions can be drawn from your data regarding the filtrate product species?
5. Write a chemical equation which represents the reaction and is supported by your data

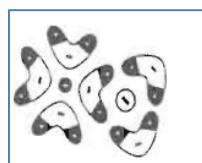
### PART 2.A. *Is Precipitation Predictable?*



In this experiment you will be asked if precipitation of a salt is a predictable event? For example, is precipitation of a salt predictable from structural characteristics of its cation such as its charge or ionic radius and/or from the position of its element in the periodic table? If you determine that, tendency to precipitate and cation structure is linked, you should be able to correctly make precipitation predictions about untested salt samples. You should also be able to identify an unknown salt based on its precipitation behavior.

### a. Information

When a salt dissolves, the partial positive and negative charges of water molecules provide a substitute for the charges (ions) in the solid salt. The negative poles of some water molecules attract the positive ions (cations) while the positive poles of some water molecules attract the negative ions (anions) in the solid salt (see figure on the right). This water-ion attraction cloaks each ion on the surface of the crystal with water molecules, and the ions are pulled into the water phase. The independent ions, now sheathed in water molecules, are free to move about in the water.



In a precipitation reaction, the process is reversed. Thus dissolving and precipitation are opposing processes. The more soluble a salt is, the less likely it will precipitate. Similarly, a salt that readily precipitates must not be very soluble.

Teams are to gather information about the solubility and precipitation behavior of a group of cations (Group I or II below -instructor assigned). In order to ensure that you are looking at cation (and not anion) effects every team will use the nitrate salts of all of the varying cations

**Cation Groups (0.1M nitrate salts): Skip  $Hg^{2+}$  &  $Cd^{2+}$ , then use  $Mn^{2+}$  instead of  $Li^+$  as shown in Table 1 later.**

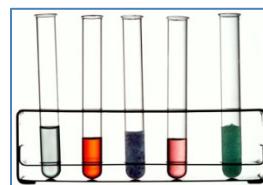
I	$Na^+$	$Ba^{2+}$	$Mg^{2+}$	$Co^{2+}$	$Ni^{2+}$	$Cu^{2+}$	$Hg^{2+}$	$Al^{3+}$	$Pb^{2+}$
II	$K^+$	$Li^+$	$Ca^{2+}$	$Sr^{2+}$	$Cr^{3+}$	$Fe^{3+}$	$Zn^{2+}$	$Ag^+$	$Cd^{2+}$

### Anion Groups (0.1M sodium salts)

In order to ensure that you are looking at anion (and not cation) effects every team will use sodium salts of tested anions.

#### b. Notes to the Procedure

##### Very much the same as c. Procedure except as following:



- a. Each group will test precipitation of either cations (I) or cations (II) **but not both** (procedure says both). Either cations (I) or (II) tested with all the **6 anions** as shown by the procedure. Groups must alternate using either cations (I) or (II) but share the results with the other group (but not the rest of the groups). For example Group #1 may select cations (I) to test, then Group #2 must select cations (II). Both Groups must accumulate and share the observation data for cations (I) & (II) and make it part of their discussions, but no need to tabulate the other group data on their report. So each group will submit their own experimental results for either cations (I) or cations (II) but not both.
- b. The procedure shows 9 cations for (I) or (II). For safety reasons you will test 8 cations of (I) or 8 cations of (II), omitting  $Hg^{2+}$  &  $Cd^{2+}$  from all parts of the experiment. Also  $Li^+$  is replaced with  $Mn^{2+}$ . Each cation in (I) or (II) is to be **tested once**. So each student in a group should test **two different cations** to precipitate, a total of **8 cations per group**.
- c. Students must prepare master table reflecting the instructions, share the results within the group and within the next group. You must show on your own table the precipitate color if any, solution color & write no precipitate (**no ppt**) if there is none for specific samples (see  $Ag^+$  example in Table 2). The procedure has the list of the cations and anions to be tested and has an example (omitting  $Hg^{2+}$  &  $Cd^{2+}$  and replacing  $Li^+$  with  $Mn^{2+}$  from all parts of the entire experiment).
- d. **Steps 1 thru 7** remain the same as the procedure. However, in **Step 4**, you will not need to use a disposable pipet because the solution agents are housed in bottles that have an eye dropper-like-lid ready to drop the agents (**only use 2 drops from each tested agent bottle per tested sample**). In **Step 6**, avoid repeating test results unless results are erroneous and the instructor authorizes you to do so and to minimize chemical waste. Work diligently.

- e. Complete the Data Analysis 1 & 2 but skip the Optional Points to Consider section.

**Table 1: CATION GROUPS TO PRECIPITATE** (0.10 M nitrate salts for each cation).

GROUP I	Na <sup>+</sup>	Ba <sup>2+</sup>	Mg <sup>2+</sup>	Co <sup>2+</sup>	Ni <sup>2+</sup>	Cu <sup>2+</sup>	Al <sup>3+</sup>	Pb <sup>2+</sup>
GROUP II	K <sup>+</sup>	Mn <sup>2+</sup>	Ca <sup>2+</sup>	Sr <sup>2+</sup>	Cr <sup>3+</sup>	Fe <sup>3+</sup>	Zn <sup>2+</sup>	Ag <sup>+</sup>

**Table 2: ANION GROUPS PRECIPITATING\* (PPT) REAGENTS:** (0.10 M sodium salts for each anion). Select cations group I or group II but not both, then add your observations on the table according to instructions & using group II example for Ag<sup>+</sup> (caution: colors may not be as shown). Compare your results with the other group I/II. Clear means no precipitate (ppt).

CATION GROUP I 2drops	CATION GROUP II 2drops	REF 2drops	Cl <sup>-</sup> 2drops	CrO <sub>4</sub> <sup>2-</sup> 2drops	I <sup>-</sup> 2drops	C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> 2drops	S <sup>2-</sup> 2drops	SO <sub>4</sub> <sup>2-</sup> 2drops	SPCTR GROUP I	SPCTR GROUP II
Na <sup>+</sup>	K <sup>+</sup>	clear								
Ba <sup>2+</sup>	Mn <sup>2+</sup>	clear								
Mg <sup>2+</sup>	Ca <sup>2+</sup>	clear								
Co <sup>2+</sup>	Sr <sup>2+</sup>	clear								
Ni <sup>2+</sup>	Cr <sup>3+</sup>	clear								
Cu <sup>2+</sup>	Fe <sup>3+</sup>	clear								
Al <sup>3+</sup>	Zn <sup>2+</sup>	clear								
Pb <sup>2+</sup>	Ag <sup>+</sup>	clear	White ppt↓	Brown ppt↓	Yellow ppt↓	White ppt↓	Black ppt↓	White ppt↓		

\*precipitation reagents are 0.10 M sodium salts of the anion.

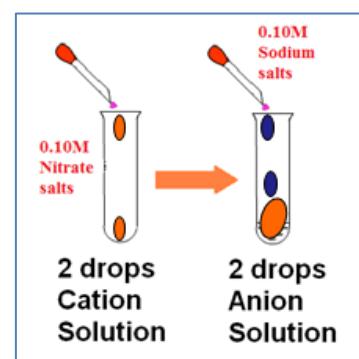
### c. Procedure

- Record a hypothesis regarding the question "is precipitation predictable?" Make sure your hypothesis is precise and relates precipitation to some cation characteristic. For example,

Your hypothesis might include "the [greater/less] the [characteristic of a cation that can be determined from the position of its element in the periodic table], the greater it's tendency to precipitate".

If your hypothesis is correct which four of your nine assigned cations are less likely to precipitate?

- Use Table 2 above to record your results (or if you want you may prepare a page sized table (8.5 x 11") that includes seven columns (one reference column for your assigned cations. and one column for each precipitating reagent), and nine blank rows. Make a copy of your form.)
- Identify the spectator ions (cation and anion) that are common to all your assigned (cation and anion) group reagents.



4. Place your data table on a flat surface and cover it with a clean plastic sheet (a heavy plastic sheet such as an overhead transparency works well).

**CAUTION**

Do not dump any of the reagents down the sink!

Discard the waste in an appropriate waste container!

Do not allow the solutions to come in contact with your skin! Wear gloves!  
(Silver ion,  $\text{Ag}^+$ , will discolor your skin. Some ions are toxic).

Add two drops of your first cation solution (Cation Group I: start with 0.1M sodium nitrate,  $\text{NaNO}_3$  or Cation Group II: start with 0.1M potassium nitrate,  $\text{KNO}_3$ , as assigned) to each column of the first cation row. Do the same for the other rows, substituting the appropriate cation solution.

5. Do not add any precipitating reagent to the first column (REF 2 drops: reference column). To each cation solution in the second column add two drops of 0.1M sodium chloride,  $\text{NaCl}$ . Add 2 drops of one of the other 5 precipitating reagents to each cation solution in the appropriate column. Do not touch the tip of the eye dropper to the cation drops or you will contaminate the precipitating agent!
6. Repeat any tests until you reach a team consensus. Record your observations (above table). Has a precipitate (ppt.) formed? Indicate "ppt↓", "none" or "unsure". Record the ppt. color. Do not discard your data table! Save your data table to refer to while conducting **Part I.B**.
7. Optional: Enter your team's data into the class data base (use a computer if available). Obtain a copy of the summarized class data and record your analysis of the data (see directives below).

#### d. Data analysis

1. Obtain the class data. Compare the tendency to precipitate of cations from elements in families 1, 2, and 12 of the periodic Table. Enter the class data results into the periodic table below.



	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1 H														
3 Li	4 Be													
11 Na	12 Mg												13 Al	
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga		
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In		
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl		

Is there an observable pattern in the data? Take care to consider that the number of reported tests for each cation or number of tested cations per family may not be the same

- Does the class data validate or dispute your recorded hypothesis regarding precipitation? Why?

#### e. Optional Points to Consider (skip this part)

- Is there a relationship between color of the salt solutions and placement of the ion's element in the periodic Table? (You might begin by marking all elements with colored cations.)
- Is there a relationship between color and tendency to precipitate? Compare the solubility of colored and colorless ions.
- Compare anion solubility. Is it possible to create a generalized rule for the solubility behavior of anions? For example, "all sulfates are soluble except \_\_\_\_."
- How do the solubility of the alkaline earth (family IIA) compounds compare? (Organize the class data graphically.) Imagine that you have a sample containing mixture of alkaline earth cations. Is it possible to separate the individual ions by selective precipitation? How?
- Is there a relationship between cation solubility and structural features such as ionic radius and/or cation charge?

#### PART 2.B. Can I Identify it? (skip this part)

Skip this Part 2B because most of the cations are already to be tested in the previous steps. Your task is to use your results and the combined results of all teams from **Part 2A** to design an experiment to identify cations in solution based on precipitation observations. You will be given a sample of well water. It contains one of the following ions:  $\text{Pb}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{K}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ca}^{2+}$ , or  $\text{Sr}^{2+}$ . Devise a way to use the precipitating agents tested in this experiment (Part 2A) to determine which ion is present record your procedure and results.

### PART 3. Concentration and Precipitation.

Your task is to investigate precipitation of ions at varying concentrations. Is there some minimum concentration of ions required for precipitation? Can toxic ions be completely removed from water by precipitation?



**Dynamic equilibrium:** rate of crystallization = rate of dissolving

#### a. Information

Each team is to investigate a specific precipitation reaction (instructor assigned). Varying team results will be shared and the resulting data bank used for analysis of results.

#### b. Notes to the Procedure

**Very much the same as procedure except as following:**

Your task is to investigate precipitation of ions at varying concentrations:

**Is there some minimum concentration of ions required for precipitation?**

**Can toxic ions be removed from water by precipitation?**



Before Reaction



After Reaction

Use the same instructions as the procedure except with few minor changes that the instructor has to approve your plan of work before starting the experiment. Four students/group need to design their own concentrations of select solutions that are shown by the first column of Table 3 below (Reactions # I through # VI (six reactions)). Then follow the instructor's clarifications of the manual instructions briefly described below:

- a. Each group must select only three types of reactions from the left column of Table 3 and carry on the experiment using the corresponding reactants shown by the rows of Table 3.
- b. Each reaction will be tested mostly with three or two different concentrations of reactants as directed by your instructor and according to the lab inventory availability of the reagents. Typically you will start reactions using 1 M reactants then dilute down reactants to 0.1 M & 0.01 M concentrations.
- c. The TA's will instruct you how to work together and approve the design of your own select reactions. A group will end up doing either 8 or 9 different reaction trials, depending on dilution limits, so each student will do at least 2 different reactions. Each group designs the reactants to differ by composition and by concentrations. Here groups should alternate reactions and compare results with each other.
- d. For many of the reagents you may have to dilute with de-ionized water to reduce (vary) concentrations using your skills ( $C_1 \times V_1 = C_2 \times V_2$ ). Stock reagents are mostly available

- a 1M solutions or 0.1M, check with the instructor for availability but each group must test 8 samples.
- e. Add only 2 drops of each the reactants for each of the reactions and do not waste chemicals.
  - f. Table 4 and Table 5 may be used as an example to design your own reactions as described above. Report your observations of the reactions to your instructor and complete your report information as instructed by the lab manual.
  - g. The instructor will guide you through next sections: *c. Procedure* (steps 1-3), *d. Additional Information* (steps 4&5), *e. Data Analysis* (steps 6 & 7). **Skip: f. Extensions** (8-10), and **Skip: g. Optional Points to Consider** (steps 11&12).
  - h. *Be diligent to safe chemicals & time. Do not repeat trials without the permission of instructor.*

**CAUTION:** Do not dump any of the reagents down the sink! Discard the waste in an appropriate waste container under the supervision of your instructor! Reduce waste by working diligently and do not repeat experimental trials without the approval of the instructor. Do not allow solutions to come in contact with your skin! Wear gloves & goggles! (Silver ion,  $\text{Ag}^+$ , will color your skin. Some ions are TOXIC).

**TABLE 3:** Team reactions (some reactants are different than those used in the UM lab manual because of the availability of inventory).

REACTION #	REACTANT #1	REACTANT #2
I	$\text{Pb}(\text{NO}_3)_2$	KI
II	$\text{Pb}(\text{NO}_3)_2$	NaOH
III	$\text{AgNO}_3$	KI
IV	$\text{ZnSO}_4$	NaOH
V	$\text{CaCl}_2$	$\text{K}_2\text{C}_2\text{O}_4$
VI	$\text{CaCl}_2$	NaOH

**TABLE 4:** Example of reactions that may be selected by a particular group of your class, say Group # 5.

REACTION #	REACTANT #1	REACTANT #2	OBSERVATIONS
II-1	0.10M $\text{Pb}(\text{NO}_3)_2$	0.10M NaOH	
II-2	0.10M $\text{Pb}(\text{NO}_3)_2$	1.0M NaOH	
II-3	0.01M $\text{Pb}(\text{NO}_3)_2$	0.01M NaOH	
IV-1	0.10M $\text{ZnSO}_4$	0.10M NaOH	
IV-2	0.10M $\text{ZnSO}_4$	1.0M NaOH	
IV-3	0.01M $\text{ZnSO}_4$	0.01M NaOH	
V-1	0.10M $\text{CaCl}_2$	0.10M $\text{K}_2\text{C}_2\text{O}_4$	
V-2	0.01M $\text{CaCl}_2$	0.01M $\text{K}_2\text{C}_2\text{O}_4$	

**TABLE 5:** Another example of alternative reactions that may be selected by next group of your class.

REACTION #	REACTANT #1	REACTANT #2	OBSERVATIONS
I-1	0.10M Pb(NO <sub>3</sub> ) <sub>2</sub>	1.0M KI	
I-2	0.10M Pb(NO <sub>3</sub> ) <sub>2</sub>	0.10M KI	
I-3	0.01M Pb(NO <sub>3</sub> ) <sub>2</sub>	0.01M KI	
III-1	0.10M AgNO <sub>3</sub>	1.0M KI	
III-2	0.10M AgNO <sub>3</sub>	0.10M KI	
III-3	0.01M AgNO <sub>3</sub>	0.01M KI	
VI-1	0.10M CaCl <sub>2</sub>	0.10M NaOH	
VI-2	0.01M CaCl <sub>2</sub>	0.01M NaOH	

### CAUTION

Do not dump any of the reagents down the sink!

Discard the waste in an appropriate waste container!

Do not allow solutions to come in contact with your skin!

### c. Procedure

1. In a small test tube add 10 drops of reactant #1 to reactant #2 (both reactants at 0.10M). Shake well. Record the amount of precipitate as "lots", "slight", "none, or "unsure". Identify the reacting ions and products. Label and save the precipitated mixture.
2. Discuss and record your team's hypothesis about concentration and precipitation. What do you expect to observe as you repeat the reaction at higher and lower ion concentrations?
3. Repeat the reaction using both reactants at equal concentrations above and below 0.10M. Record a team consensus about the amount of ppt as "lots", "slight", "none", or "unsure". Label and save any precipitated mixture.



### d. Additional Information

If < 0.10M reagent is not available, dilute the 0.10M sample. (for example, 2 drops of 0.10M reagent + 18 drops of distilled water = 0.010M)

If > 0.10M reagent is not available, add a few crystals of the solid to its 0.10M reagent.

4. (**skip this part**) Filter any precipitated mixture from steps 1, 3, or 4 and collect the filtrate (the clear liquid passing through the filter). Divide the filtrate into two portions. (Note: if additional filtrate is needed you may repeat a reaction using proportionately larger volumes of each reagent.) To one filtrate portions add a few crystals of any of the reactant #1 chemical. To the other portion add 1 to 5 drops of 0.1M Na<sub>2</sub>S. (Note: sulfide salts of most transition and heavy metal ions are insoluble) does a precipitate form? Does the filtrate appear to contain unprecipitated ions?

5. Enter your team's data into the class database (use a computer if available). Obtain a copy of the summarized class data and conduct an analysis of the data (see directives below).

**e. Data analysis: Use Table 3, Table 4 & Table 5 to record your results**

6. Obtain the class data do you observe patterns in the data regarding concentration effects and precipitation? For example, does precipitation of all salts occur within the same concentration values? Does the amount of precipitate increase or decrease with increase in concentration? Are all ions removed from solution upon precipitation?
7. Does the data support or contradict your hypothesis regarding concentration and precipitation? Explain your reasoning.

**f. Extensions (skip this part)**

8. What will you observe if the reactants are not equal in concentration? specifically, what will happen to the amount of precipitate formed, if the amount of reactant 2 is at a concentration greater than 1.0M while reactant #1 remains at 0.1M concentration?
9. Does the order of addition of reactants effect the results? for example, add  
5 drops of 0.1M Pb(NO<sub>3</sub>)<sub>2</sub> to 5 drops of 1M NaOH →?  
5 drops of 1M NaOH to 5 drops of 0.1M Pb(NO<sub>3</sub>)<sub>2</sub> →?
10. Add 3mL of water to the middle of beaker or petri dish so a pool is formed in the center. Simultaneously place crystals (or drops) of each reactant at opposite sides of the pool. Do not move, shake or stir the contents. Observe for several minutes. Now repeat the procedure except wait several minutes before adding the second reactant. Compare the results.

**g. Optional Points to Consider (skip this part)**

11. Is there a minimum concentration of ions required for precipitation?
12. You need to remove an "undesirable ion" from solution. Is it possible to completely remove the ion from solution by precipitation and filtration? Does the identity and concentration of the precipitating agent matter?

**PART 4. Solvent Pollution & Precipitation**

A drum containing a solvent other than water is accidentally dumped into a pond of water containing dissolve salts. What, if anything, will happen? In inquiry you will compare the solubility and precipitation of salts in different solvents (water, acetone and, hexane).

**a. Information**

Water is polar; Acetone (CH<sub>3</sub>COCH<sub>3</sub>) is moderately polar; hexane (C<sub>6</sub>H<sub>14</sub>) is nonpolar.

Your team should investigate the same reactants assigned in **Part 3**.

You will need 5-6 crystals of each assigned reactant

You will need about 4ml (80 drops) of each solvent

## b. Notes to the Procedure

Very much the same as procedure except as following:

Follow the instructor's directions that he will guide you through the procedure shown in the procedure next, and he may ask you to skip some steps (SKIP STEP 9). Group work: for each reaction (in water or in acetone or in hexane) each group tests the solubility of two solids separately ( $\text{CaCl}_2$  &  $\text{K}_2\text{C}_2\text{O}_4$ ), where each two students of the group test one solid while the other two students test the other alternative solid. Complete Table 6 for each group results.

**TABLE 6: Solids Solubility in Polar & Non-Polar Solvents (total 6 samples to test)**

Solid Type	Ionized Water	Acetone	Hexane
I. $\text{CaCl}_2$			
II. $\text{K}_2\text{C}_2\text{O}_4$ (potassium oxalate)			
Supernatant (I + II)			

## c. Procedure

1. Record your hypothesis regarding effect of varying solvent polarity on solubility and precipitation of salts. If your hypothesis is correct what will you observe? For example, will precipitation occur in all solvents? Will the amount of precipitate differ?
2. Test and compare the solubility of your assigned salt reactants in water, acetone, and hexane.

GROUP WORK: FOR EACH REACTION (IN WATER OR IN ACETONE OR IN HEXANE) EACH GROUP DO 2 TRIALS, EACH 2 STUDENTS DO 1 TRIAL

1. DO THE REACTION WITH WATER

$\text{CaCl}_2$      $\text{K}_2\text{C}_2\text{O}_4$

$\text{CaCl}_2(s)$  + 2mL  $\text{H}_2\text{O}$

$\text{K}_2\text{C}_2\text{O}_4(s)$  + 2mL  $\text{H}_2\text{O}$

MIX

ppt?



### CAUTION

Acetone and hexane are volatile!

Do not inhale acetone or hexane keep the samples closed or use a hood!

Do not dump acetone or hexane down the sink!

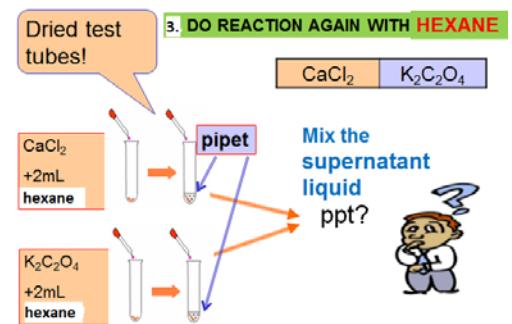
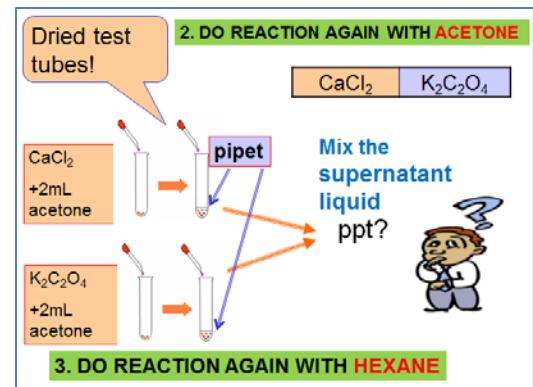
Discard the acetone or hexane in an appropriate (organic) waste container!

Add a few crystals of reactant #1 to each of three separate dry test tubes labeled water, acetone or hexane. Add= 2.0mL (40 drops) of water or acetone or hexane to the crystals.

Mix vigorously. Record your observations. Are the crystals “insoluble”, “slightly soluble” or “soluble” in each testing solvent? Save the labeled samples for later use.

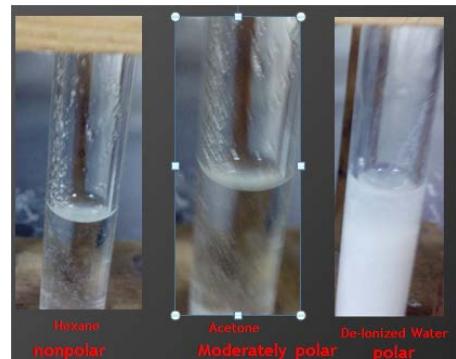
Repeat the tests with reactant #2. Save the labeled samples for later use.

- Separate any undissolved solid from the solvent solution (containing dissolved crystal). For example, pipet or decant off the supernatant liquid (the clear solution above any undissolved solid) into clean and dry test tubes or containers (labeled to identify the contents).
- Test and compare the effect of solvent environment on precipitation. Combine the clear liquids of reactant #1 and #2 from the same solvent mixture (e.g. water with water or acetone with acetone or hexane with hexane). Does precipitation occur? How much? Record "lots", "slight", "none", or "unsure", repeat any tests until you reach a consensus.
- Do your team results show any pattern between precipitation (step 4 above) and solubility (step 3) of the salts in a given solvent
- Enter your team's data into the class database (use a computer if available), obtain a copy of the summarized class data and conduct an analysis of the data (see directives below)



#### d. Data Analysis

- Are any visible patterns in the class data regarding effect of solvent polarity on solubility and precipitation of salts? Record any data patterns and give specific examples.
- Does the class data confirm or deny your recorded hypothesis? How do you know?



#### e. Optional Points to Consider (skip this part)

- The concentration of NaCl in Utah's great salt lake is = 6M. If a drum containing acetone accidentally spills into the lake is it likely that some NaCl will precipitate? Information: the solubility of NaCl = 35.7g/100cc (0°C) or 39.1g/100cc (100°C).

## **PART 5. Can I purify it? (skip this part)**

Your task is to use your acquired knowledge and skills to design an experiment to selectively remove ions from solution by precipitation and filtration. Each group will do 2 samples where each 2 students will do 1 sample (once each). Follow the instructor's directions that he will guide you through the following procedure: The water effluent from a plant contains a mixture of Cu<sup>2+</sup>, Fe<sup>3+</sup>, and Na<sup>+</sup> ions. You are to design a method for maximizing removal of Cu<sup>2+</sup> ions from the mixture. You may use any of the reagents (precipitating agents or solvents) tested in this experiment and/or filtration. Note: if you are successful the blue color of Cu<sup>2+</sup>(aq) will fade. The presence of copper (II) ions can be checked quantitatively. The sample's absorbance can be read at  $\lambda_{\text{optimum}}$  ( $\lambda$  650) using a spectrophotometer (Appendix A4). A reading of zero indicates removal of the Cu<sup>2+</sup> ions.

*Collect all your results as instructed on the first page of this experiment and complete your final lab report by adding and answering the next section VII. Then hand deliver the report to your instructor. No reports will be accepted after the end of this lab session.*  
*Prepare all needed tables and report format ahead of the lab session so you can be ready to enter & share collected data.*

**RECORD YOUR ENTIRE RAW DATA ON THE DATASHEET OF PAGE 17. THEN CONTINUE TO FINISH ALL THE NEXT SECTION “VII. REPORTS OF RESULTS”.**

VC211 EXPERIMENT E5 DATASHEET: PRECIPITATION & WATER PURITY													
STUDENT:		ID:		SECTION#:		TA:							
GRP#:													
<b>PART 1:</b> What is a precipitate. Each 2 students tests 1 sample once													
	CuSO <sub>4</sub> 5mL (0.1M) Color	BaCl <sub>2</sub> 5mL (0.1M) Color	CuSO <sub>4</sub> + BaCl <sub>2</sub> Color	ppt↓ Yes/No	<b>Filtrate observed property</b>								
2students													
2students													

**PART 2.A.** Is Precipitation Predictable? Group efforts, test only Group I or Group II as assigned, 2 raw reactions/student. Clear = means no precipitate, then record solution color.

CATION GROUP I	CATION GROUP II	REF WATER	Cl <sup>-</sup> 2drops	CrO <sub>4</sub> <sup>2-</sup> 2drops	I <sup>-</sup> 2drops	C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> 2drops	S <sup>2-</sup> 2drops	SO <sub>4</sub> <sup>2-</sup> 2drops	SPECTATOR IONS
Cations no. drops →		2drops	2drops	2drops	2drops	2drops	2drops	2drops	GROUP I    GROUP II
Na <sup>+</sup>	K <sup>+</sup>	clear / colorless							
Ba <sup>2+</sup>	Mn <sup>2+</sup>	clear / colorless							
Mg <sup>2+</sup>	Ca <sup>2+</sup>	clear / colorless							
Co <sup>2+</sup>	Sr <sup>2+</sup>	clear / colorless							
Ni <sup>2+</sup>	Cr <sup>3+</sup>	clear / colorless							
Cu <sup>2+</sup>	Fe <sup>3+</sup>	clear / colorless							
Al <sup>3+</sup>	Zn <sup>2+</sup>	clear / colorless							
Pb <sup>2+</sup>	Ag <sup>+</sup>	clear / colorless	White ppt↓	Brown ppt↓	Yellow ppt↓	White ppt↓	Black ppt↓	White ppt↓	

**PART 3. Conc. & Precip.: Each team uses Table 3 & design different reactions than Table, minimum 2 reactions per student**

TABLE 5: RECORD YOUR RAW DATA HERE similar to Table 4. Add only 2 drops of each reactant				TABLE 3: DESIGN REACTIONS FROM HERE			TABLE 4: SAMPLE REACTIONS DESIGN			
REACTION #	REACTANT #1 & CONC	REACTANT #2 & CONC	OBSERVATIONS	REACTION #	REACTANT #1	REACTANT #2	Reactant #	REACTANT T #1 & Conc.	REACTANT T #2 & Conc.	OBSERV.
				I	Pb(NO <sub>3</sub> ) <sub>2</sub>	KI	II-1	0.10M Pb(NO <sub>3</sub> ) <sub>2</sub>	0.10M NaOH	
				II	Pb(NO <sub>3</sub> ) <sub>2</sub>	NaOH	II-2	0.10M Pb(NO <sub>3</sub> ) <sub>2</sub>	1.0M NaOH	
				III	AgNO <sub>3</sub>	KI	II-3	0.01M Pb(NO <sub>3</sub> ) <sub>2</sub>	0.01M NaOH	
				IV	ZnSO <sub>4</sub>	NaOH	IV-1	0.10M ZnSO <sub>4</sub>	0.10M NaOH	
				V	CaCl <sub>2</sub>	K <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	IV-2	0.10M ZnSO <sub>4</sub>	1.0M NaOH	
				VI	CaCl <sub>2</sub>	NaOH	IV-3	0.01M ZnSO <sub>4</sub>	0.01M NaOH	
							V-1	0.10M CaCl <sub>2</sub>	0.10M K <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	
							V-2	0.01M CaCl <sub>2</sub>	0.01M K <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	

**PART 4. Solvent Pollution & Preci.: Group efforts, each 2 students study solubility of 1 solid**

TABLE 6: Solids Solubility in Polar & Non-Polar Solvents (total 6 samples to test)

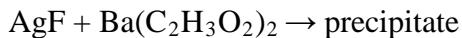
Solid Type (thoroughly dry inside test tubes)	Ionized Water 2mL	Acetone 2mL	Hexane 2mL	Note: Use the solubility table from CH 4, VC210 to predict if precipitate is formed from mixing the supernatants
I. CaCl <sub>2</sub> <0.2g				
II. K <sub>2</sub> C <sub>2</sub> O <sub>4</sub> <0.2g				
Supernatant (I + II)				

## VII. REPORTS OF RESULTS

**Students must print/copy/type/paste this entire section (as is) and then handwrite the answers that only relevant to your experiment while ignoring the other questions that are not relevant to the E5. Provide additional spaces as needed. The last “Grade” sheet is for the instructor to complete but you must print the form for him and include within this section**

### Pre-Laboratory Report

1. Name: \_\_\_\_\_ Team#: \_\_\_\_\_ Date: \_\_\_\_\_ Section: \_\_\_\_\_
2. when a solution of silver fluoride, 0.1M AgF, is mixed with a solution of barium acetate, 0.1M Ba(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> a precipitate forms:



- A. In order to identify the precipitating ions you conduct some experimental tests.
  - 1) you substitute a solution of 0.1M sodium acetate, NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>, for the Ba(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> solution and mix it with 0.1M AgF
$$\text{AgF} + \text{NaC}_2\text{H}_3\text{O}_2 \rightarrow ?$$
What is the purpose of this test?
  - 2) What do the test results bellow tell you about the AgF + Ba(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> reaction?
$$\text{AgF} + \text{Ba}(\text{NO}_3)_2 \rightarrow \text{precipitate*}$$

\* The precipitate that forms is identical in properties to that formed in the AgF + Ba(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> reaction.

- 3) Which reference blank test (indicated below) did you use to directly test the hypothesis that silver ions are critical to precipitate formation?

**Circle** the reference blank test reagent combination that could be used to directly verify if precipitate formation involves silver ions.

AgF + Ba(NO<sub>3</sub>)<sub>2</sub>

NaF + Ba(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>

AgF + KC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>

3. Prepare a data table for **Part 2.A** (of the experimental) on an 8 1/2× 11 sheet of paper and make a copy to place under the plastic sheet. Note: your instructor will assign you to one cation group to study in lab. In the meantime leave the cation column blank.

4. Record a precise hypothesis and expected observations for Part 2a for suggestions regarding formulating a hypothesis.

**Team Report:** Entire team collaborate & hand write discussions in provide spaces

Must be submitted in team leader ALR report, then on your individual report write across the pages of the Team Report "SEE TEAM LEADER REPORT name of leader xxxx". This saves time in copying same information

Name: \_\_\_\_\_ Team#: \_\_\_\_\_ Date: \_\_\_\_\_ Section: \_\_\_\_\_

### **Part I. What is the precipitate?**

Observations of reactants and product mixture

CuSO <sub>4</sub>	BaCl <sub>2</sub>	Product Mixture

Possible reacting ions:

Experimental tests to identify reacting ions

Filtrate observations

### **Analysis and Conclusion**

Identity of reacting ions:

Proposed products and properties listed in CRC Handbook or from any available data source:

Chemical equation based on data:

### **Part 2. Precipitation Studies**

#### **Part 2.A. Is precipitation predictable?**

##### **Team hypothesis and expected observations:**

Four cations least likely to precipitate:

Spectator ions (cation and anion) common to all assigned reactions?

##### **Team Observations**

PRECIPITATING REAGENTS*						
CATION REF	Cl <sup>-</sup>	CrO <sup>2-</sup> <sub>4</sub>	I <sup>-</sup>	C <sub>2</sub> O <sup>2-</sup> <sub>4</sub>	S <sup>2-</sup>	SO <sub>4</sub> <sup>2-</sup>

\*precipitating reagents are 0.1M sodium salts of the anion.

## Data Analysis (Class Data)

Attach a copy of the class data your team used for the purpose of data analysis.

Compare the solubility or tendency to precipitate of the cations with elements in families 1, 2, and 12 in the periodic table. Is there an observable pattern? Record your analysis below. **Be sure to refer to the attached class data to support any conclusion.**

Note: the number of reported tests for varying cations may not be the same. The comparative percentage of precipitates (out of total tests) per cation is more valid than number of precipitates reported. Also note that the number of tested cations per family may not be the same.

### Precipitation vs Cation family ( 1 vs. 2 vs 12)

#### Part 2.B. Can I identify it?

Unknown sample # \_\_\_\_\_

### Part 3. Water Purity and Concentration Studies

Assigned reactants: # 1 \_\_\_\_\_ #2 \_\_\_\_\_

#### **Observations of reactants and products**

Reaction #1	Reactant #2	Precipitate ("Lots", "Slight", "none", "Unsure")
Identity of reacting ions	Identity of Participate	

#### **Team Hypothesis and expected observations:**

#### **Experimental tests and observations:**

Precipitate yield\* vs Concentration Reactants

= and <0.1M	= and at 0.1M	= and >0.1M

\*Record ("lots". "slight". "none", "unsure")

#### **Filtrate observations (skip this part)**

Filtrate	Filtrate+Crystals	Filtrate+0.1M Na <sub>2</sub> S
Skip this part	Skip this part	Skip this part

### **Analysis of team results**

1. What do your team results indicate about concentration and precipitation?
2. Does the filtrate contain un-precipitated reactant ions? How do you know?

### **Analysis of Class Data**

1. What results are shown in the class data? Use the class data to give specific examples of any patterns.
2. Does the class data validate your hypothesis? Explain.

### **Part 4. Solvent Pollution and Precipitation**

#### **Team hypothesis and expected observations:**

Assigned Reactants: Reactant #1= \_\_\_\_\_ Reactant #2 =\_\_\_\_\_

#### **Team Observations:**

##### **Solubility and Precipitation of Reactants in Solvents of Varying Polarity**

Solvent	Solubility Reactant #1	Solubility Reactant #2	Precipitate (“Lots”, “Slight”, “none”, “Unsure”)
H <sub>2</sub> O			
Acetone CH <sub>3</sub> COCH <sub>3</sub>			
Hexane C <sub>6</sub> H <sub>14</sub>			

#### **Data Analysis (class data)**

1. Record any patterns shown in the class data regarding effect of solvent polarity on solubility and precipitation of salts. Give specific examples referring to the class data.
2. Does the class data validate or refute your team hypothesis? Explain your reasoning.

### **Part 5. Can I purify it? SKIP THIS PART**

Unknown sample # \_\_\_\_\_

## Laboratory Discussion

1. Is it possible to predict if a precipitate will be white or a color other than white based on the position of the cation's element in the periodic table? Organize the class data (**Part 2A**) in a graph to answer this question.

Based on your answer predict the compound within each pair that is a color other than white: nickel sulfate or lead sulfate; cobalt oxalate or strontium oxalate?

2. Is there a relationship between precipitation and ion charge? For example, what generalizations can be made about the solubility of ionic compounds, if both the cation and anion are singly or multiply charged? Organize the class data (**Part 2A**) in a graph to answer this question.

Based on your answer select the compound within each pair that is less soluble in water: iron (III) chloride,  $\text{FeCl}_3$ , or iron oxide,  $\text{Fe}_2\text{O}_3$ ; sodium oxalate,  $\text{Na}_2\text{C}_2\text{O}_4$ , or calcium oxalate,  $\text{CaC}_2\text{O}_4$

3. Is it possible to predict cation solubility based on the position of its element in the periodic table? For example, compare precipitation of family 1, 2, and 12 cations. Organize the class data (**Part 2A**) in a graph to answer this question.

Based on your answer select the compound within each pair that is less soluble in water: nickel bromide,  $\text{NiBr}_2$  or platinum bromide,  $\text{PtBr}_2$ ; zinc iodate,  $\text{Zn}(\text{IO}_3)_2$ , or mercury iodate,  $\text{Hg}(\text{IO}_3)_2$

4. Examine the relationship, if any, between concentration and the amount of precipitate. Organize the class data (**Part 3**) in the form of a histogram (if time permits during the lab session). For example, compare the amount of hydroxide precipitates of varying cations at the different concentrations. It is a scientific fact that calcium hydroxide is more soluble than zinc hydroxide which is more soluble than lead hydroxide. Does the class data support or refute these scientific facts?

You are to remove  $\text{Hg}^{2+}$  ions from a contaminated body of water by precipitation and filtration will it be possible to completely remove all  $\text{Hg}^{2+}$  ions will a particular precipitating agent remove more  $\text{Hg}^{2+}$  ions than another.

5. What is the relationship, if any, between salt solubility, precipitation, and solvent polarity? Organize the class data from **Part 4** (table or graph) to answer this question.
6. **Skip this part:** If a drum of acetone accidentally spills into Utah's Great Salt Lake is it likely that  $\text{NaCl}$  will precipitate? The concentration of  $\text{NaCl}$  in the Great Salt Lake is = 6M. The CRC Handbook reports the solubility of  $\text{NaCl}$  = 35.7g/100cm<sup>3</sup> at 0°C and 37.1 g/100cm<sup>3</sup> at 100°C.

**Team Assessment Form: Instructor may have different evaluation form**

Team name: \_\_\_\_\_

lab section: \_\_\_\_\_

Team#: \_\_\_\_\_

date: \_\_\_\_\_

Determine and record the percent contribution of each team member to the experiment.

Your name: \_\_\_\_\_

percent contribution: \_\_\_\_\_

Other team names: \_\_\_\_\_  
\_\_\_\_\_percent contribution: \_\_\_\_\_  
\_\_\_\_\_Describe your contributions to the completion of the experiment and the team report and discussion presentation. **Return this form to your instructor.****Laboratory Discussion Team Presentation  
Grading Form: SKIP THIS PART**

Team number: \_\_\_\_\_ Date: \_\_\_\_\_ Section: \_\_\_\_\_

Team members: \_\_\_\_\_

During the post-lab discussion period at the conclusion of the experiment, each team will be asked to present their answer to one of the post-lab discussion questions. These presentations will count for a total of 14 points. Points will be assigned by your instructor using the same criteria used in prior discussions (does the groups' answer utilize the class data?, is the data organized in a helpful manner?, is the answer logical based on the class data?, is the group's answer clearly communicated?, is the group's presentation well organized and effective?) and student input.

Use this sheet to assign a grade between 0 -5 for each team except for your own team. Grades should be based on the effectiveness of the overall presentation:

0	1	2	3	4	5
unsatisfactory	poor	fair	good	very good	excellent

Consider if the team's presentation is well organized and effective (is the problem clarified and the process used to determine the answer clear and logical)? Are the team's conclusions understandable and useable (e.g. could you answer an exam question related to the investigation)?

Question	Points	Comments
1		
2		
3		
4		
5		
6		

## **HOW TO SUBMIT E5 ALR REPORT?**

- 1. Teams to meet in assigned areas under the supervision of the TA.**  
You are not allowed to leave until E5 is completed, glassware and lab areas clean to original or better condition, discuss entire data and report with the TA and then submit your completed individual reports.
- 2. Each student must submit individual report**
- 3. Omit all sections that said in procedure “Omit” or faded text, such as Part 5, or when data is not available such as CRC Handbook , data bank base on compute (not available). But you must clearly mark across entire section of that part the word “OMIT”.**
- 4. The group report part is to be completed by the group and submitted to the group leader report. While the remaining members of the group will write across that page of their own team report “SEE TEAM LEADER REPORT & give his name xxxx”. This way you save time in duplicating the Team Report data.**

**Grading: SKIP THIS PAGE**

Make sure you include this form with your report for the instructor to complete

Name: \_\_\_\_\_ Team#: \_\_\_\_\_ Date: \_\_\_\_\_ Section: \_\_\_\_\_

	Possible	Points Earned
pre-lab (5 pts)		
A. Was completed correctly and turned in Laboratory*	A.5	_____
*student must be present and perform experiment to receive points.		
B. Part 1. Observations recorded (1 pt) and reasonable conclusions (based on tests) are stated (3 pts)	B.4	_____
C. Part 2. Observations recorded (1 pt); a reasonable hypothesis is recorded where identified cations conform to hypothesis (1 pt); spectator ions are identified (1 pt); reasonable conclusions (class data referred to) are stated regarding cation solubility and position in the periodic table (3 pts); hypothesis is re-examined (1 pt)	C.7	_____
D. Part 3. Observations recorded (1 pt); a reasonable hypothesis is recorded (1 pt); reasonable conclusions (based on team and class data) are stated regarding concentration vs. precipitation(4 pts)		
OR Part 4. Observations recorded (1 pt); hypothesis is reasonable(l pt); reasonable conclusions (class data is used and referred to) are given regarding solubility, ppt. and solvent polarity (4 pts)	D.6	_____
E. Part 5A. Observations recorded (1 pt); the ion is correctly identified using an appropriate scheme (3 pts).	E.4	_____
<b>lab discussion (14 pts)</b>		
F. Team presentation	F.14	_____
<b>Total</b>	40	_____

## **REFERENCES**

1. University of Michigan laboratory manual: Konigsberg Kerner & Penner-Hahn, Hayden McNeil; Hands on Chemistry Laboratory Manual, 1st Ed., Jeffrey A. Paradis, Kristen Spotz, McGraw Hill Higher Education Press, 2006 (Experiment 6- Precipitation and water Purity pages 49-73, Appendix C.1 Precipitation and Solubility pages 185-188, and Appendix A.3 Filtration page 167 ).
2. VC210 textbook: Chemistry-The Central Science (Theodore L. Brown; H. Eugene LeMay, Jr.; Bruce E. Bursten), Pearson International edition (Pearson Prentice-Hall, Inc.), 12<sup>th</sup> edition 2012, ISBN 9780321696724 (Chapter 4 Table 4.1 page 121 and Chapter 17 pages 722-739). Topics: solubility guidelines for common ionic compounds in water, solubility equilibria, factors that affect solubility, precipitation & separation of ions, & qualitative analysis of metallic elements.

### **3. ADDITIONAL REFERENCES**

**THE FOLLOWING PAGES ARE FOR YOUR REVIEW ONLY AND NO NEED TO COPY/PRINT INTO YOUR LAB REPORT. HOWEVER, YOU ARE ENCOURAGED TO REFER TO AND DISCUSS BRIEFLY IN YOUR REPORT.**

A. VC210 textbook: Chemistry-The Central Science (Theodore L. Brown; H. Eugene LeMay, Jr.; Bruce E. Bursten), Pearson International edition (Pearson Prentice-Hall, Inc.), 12<sup>th</sup> edition 2012, ISBN 9780321696724 (Chapter 4 Table 4.1 page 121)

**TABLE 4.1 • Solubility Guidelines for Common Ionic Compounds in Water**

Soluble Ionic Compounds	Important Exceptions
Compounds containing	
$\text{NO}_3^-$	None
$\text{CH}_3\text{COO}^-$	None
$\text{Cl}^-$	Compounds of $\text{Ag}^+$ , $\text{Hg}_2^{2+}$ , and $\text{Pb}^{2+}$
$\text{Br}^-$	Compounds of $\text{Ag}^+$ , $\text{Hg}_2^{2+}$ , and $\text{Pb}^{2+}$
$\text{I}^-$	Compounds of $\text{Ag}^+$ , $\text{Hg}_2^{2+}$ , and $\text{Pb}^{2+}$
$\text{SO}_4^{2-}$	Compounds of $\text{Sr}^{2+}$ , $\text{Ba}^{2+}$ , $\text{Hg}_2^{2+}$ , and $\text{Pb}^{2+}$
Insoluble Ionic Compounds	Important Exceptions
Compounds containing	
$\text{S}^{2-}$	Compounds of $\text{NH}_4^+$ , the alkali metal cations, $\text{Ca}^{2+}$ , $\text{Sr}^{2+}$ , and $\text{Ba}^{2+}$
$\text{CO}_3^{2-}$	Compounds of $\text{NH}_4^+$ and the alkali metal cations
$\text{PO}_4^{3-}$	Compounds of $\text{NH}_4^+$ and the alkali metal cations
$\text{OH}^-$	Compounds of $\text{NH}_4^+$ , the alkali metal cations, $\text{Ca}^{2+}$ , $\text{Sr}^{2+}$ , and $\text{Ba}^{2+}$

**B.** University of Michigan laboratory manual: Konigsberg Kerner & Penner-Hahn, Hayden McNeil; Hands on Chemistry Laboratory Manual, 1st Ed., Jeffrey A. Paradis, Kristen Spotz, McGraw Hill Higher Education Press, 2006 (Experiment 6- Precipitation and water Purity pages 49-73, Appendix C.1 Precipitation and Solubility pages 185-188, and Appendix A.3 Filtration page 167 ).

*Laboratory Techniques*

*Filtration*

*Appendix A*

## A. 3 FILTRATION

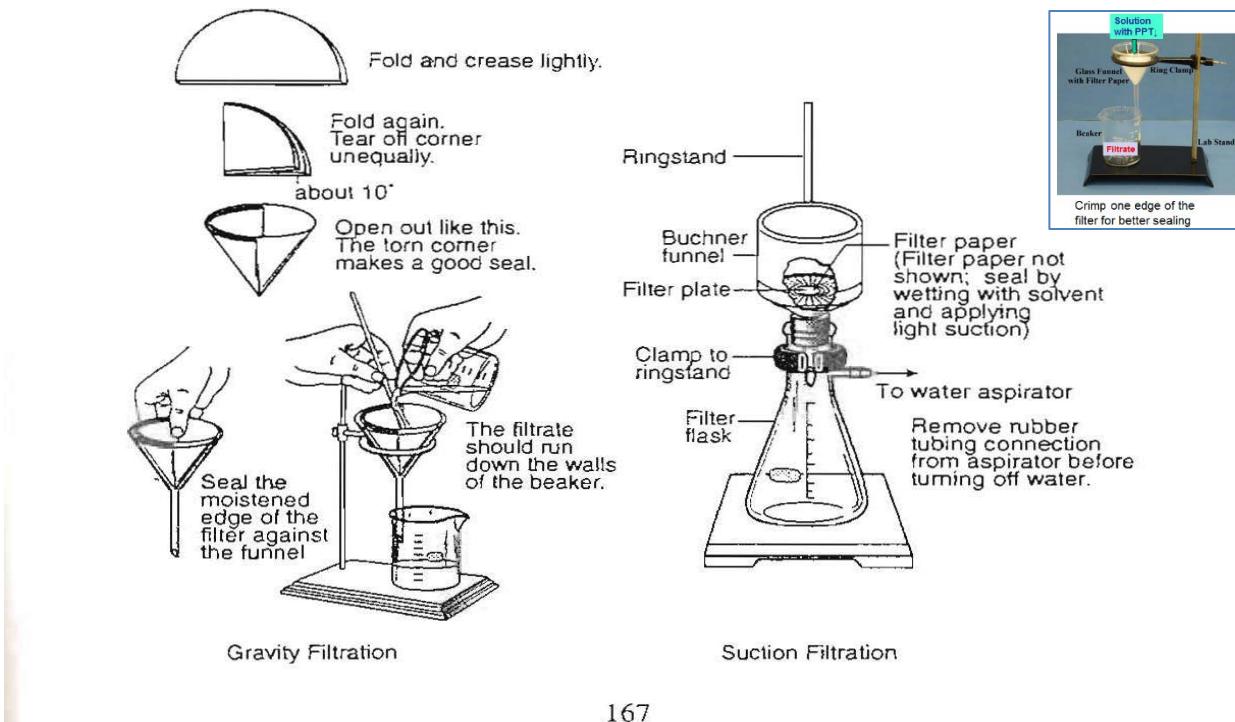
A common laboratory procedure is the separation of a solid from a liquid by filtration. In the laboratory you will choose between two methods of filtration. *Gravity filtration* is a rather slow process carried out with simple equipment. *Suction filtration* requires a water pump and special glassware, but once set up, it proceeds rapidly.

### *How to Gravity filter*

1. Set up the equipment. Fold the filter paper as shown. Place it in the funnel and moisten it with distilled water or the solvent in use to seal it against the funnel.
2. Decant (pour off) most of the supernatant solution (the clear liquid above the solid) into the funnel. Using a glass rod, pour the precipitate into the funnel as shown.
3. Wash the precipitate with small amounts of solvent to get rid of trace impurities.
4. Dry the precipitate by leaving it in the open or putting it in an oven at a suitable temperature.

### *How to Filter with a Buchner Funnel*

1. Set up the equipment and clamp the filter flask to the ringstand.
2. Place the filter paper in the funnel, and moisten it with water or the solvent to be used.
3. Turn the vacuum on. While the vacuum is on, pour into the funnel the solution to be filtered.
4. Wash the precipitate with a small amount of solvent. Allow the solid to dry by drawing air through the funnel.
5. Remove the precipitate from the filter by lifting one edge of the filter paper with a spatula.



## APPENDIX C

### CHEMICAL REACTIVITY

#### C.1 PRECIPITATION AND SOLUBILITY

Precipitation reactions are a common type of reaction involving ions in solution that react to form a solid. An ion is an electrically charged atom or radical. Ionic compounds contain a positive ion (for example,  $\text{Na}^+$ ,  $\text{Pb}^{2+}$ ) and a negative ion (for example,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ). Compounds containing a positive ion other than hydrogen and a nonmetal other than oxygen or  $\text{OH}^-$  (hydroxide) are called *salts*. Salts are solids at room temperature. Their melting and boiling points are higher than those of molecular solids due to the large amount of energy required to overcome the attractive forces between oppositely charged ions. Although salts are difficult to melt, many can be dissolved easily in water. When a salt crystal dissolves, it does not simply come apart into ions. Rather, the associated ions comprising the crystal are separated (*dissociated*) by the molecules of the water into which it is dissolved.



Breakup of a salt crystal by water

The electron pairs are shared unequally between the hydrogen and oxygen atoms in water molecules. The O end of the molecule attracts the shared electron pair more strongly than the H. The O end of the molecule is therefore negative with respect to the H end. Such a separation of charge creates an electric *dipole*. The partial positive and negative charges on water molecules provide a substitute for the positive and negative charges in the salt crystal:

- Each positively charged ion is surrounded by water molecules with their negatively charged oxygens turned toward it, and each negatively charged ion is surrounded by water molecules with their positively charged hydrogens turned toward it. The ions are said to be *hydrated* by water. The hydrated aqueous state is represented by placing (aq) as a subscript next to the ionic symbol -- for example,  $\text{K}^+_{(\text{aq})}$  or  $\text{Cl}^-_{(\text{aq})}$ . Once you dissolve the salt, it is completely ionized.

## Solubility

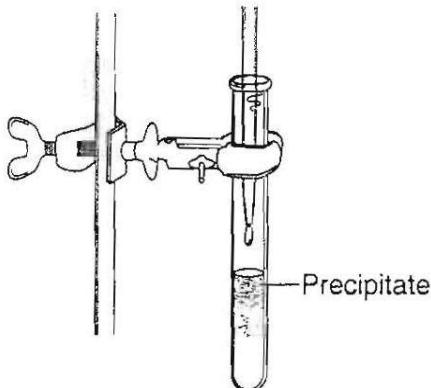
**Solubility** is defined as the amount (g or mol) of *solute* (substance you are dissolving) that will dissolve in a given quantity (volume) of solvent at a specific temperature and pressure to give a *saturated* solution. Saturated solutions contain so much dissolved substance that no more can be dissolved. For example, the *CRC Handbook* lists the solubility of table salt, NaCl, as 35.70g/100mL water at 20°C. Since the mass of a mole of NaCl = 58.44g, a saturated solution of NaCl is 6.11M or the solubility is 6.11mol/L at 20°C. This means that it is impossible to prepare an aqueous solution (solution where the solvent is water) of NaCl that is greater than 6.11M in concentration at a temperature of 20°C.

The solubilities of ionic salts vary tremendously. The solubility of HgS is  $10^{-26}$ mol/L at 20°C in water. The low solubility of HgS implies that interionic forces are greater than the force of attraction between the charged ions and the water molecules.

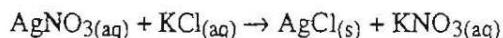
The solubility of substances are dependent on temperature and the nature of the solvent. For example, the *CRC Handbook* indicates that the solubility of NaCl in water is 35.70g/100mL water at 20°C and 39.12g/100mL at 100°C. NaCl is only slightly soluble in alcohol and is insoluble in benzene.

## Precipitation

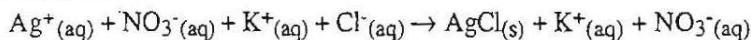
If solutions of two different salts are mixed, all of the different ions are free to interact in the resulting solution. Even though all of the ions were soluble in the two initial solutions, new ion combinations may result in an insoluble *precipitate* when the solutions are mixed. A precipitate is a solid formed in solution by a reaction. These are sometimes called metathesis



reactions, from a Greek word meaning "interchange." Ions exchange partners to achieve more favorable electrostatic interactions. For example, if you mix solutions of silver nitrate and potassium chloride, you obtain a precipitate of insoluble silver chloride:



Silver chloride,  $\text{AgCl}$ , is highly insoluble and therefore very few  $\text{Ag}^+$  and  $\text{Cl}^-$  ions remain in solution. If the solution is evaporated (after removing the  $\text{AgCl}$ ) crystals of  $\text{KNO}_3$  are produced with a negligible amount of  $\text{AgCl}$ . The ionic equation makes it easier to understand the mechanism of reaction:



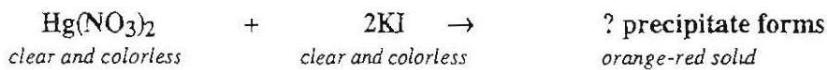
where the letters in parentheses describe the physical state of each species (s, solid; g, gas; l, liquid; aq, aqueous solution).

A *net* ionic equation includes only the ions undergoing change. Because  $\text{K}^+$  and  $\text{NO}_3^-$  appear on both sides of the equation in the same form, they can be canceled. Thus, the net ionic equation for the reaction is  $\text{Ag}^+_{(\text{aq})} + \text{Cl}^-_{(\text{aq})} \rightarrow \text{AgCl}_{(\text{s})}$

A common error in writing ionic equations is to ionize a salt into two or more parts that do not balance with the original. Thus, you might incorrectly ionize  $\text{CaCl}_2$  into  $\text{Ca}^{2+} + \text{Cl}_2$  rather than  $\text{Ca}^{2+} + 2\text{Cl}^-$ . Splitting a salt must produce positive and negative charges in equal amounts.

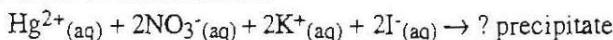
## Precipitate Identification

In order to identify the products of a reaction, scientists use experimental tests, knowledge, other information (e.g., from the *CRC Handbook*), and logic. For example, if you mix equal volumes of 0.1M mercuric nitrate and potassium iodide, you observe:



### Knowledge and Logic:

Recognize that each dissolved salt is ionized:

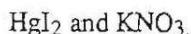


Recognize that the product must contain both anions and cations and therefore,

$\text{Hg}^{2+}_{(\text{aq})} \text{K}^+_{(\text{aq})}$  does not form the precipitate  
 $\text{NO}_3^-_{(\text{aq})} + \text{I}^-_{(\text{aq})}$  does not form the precipitate

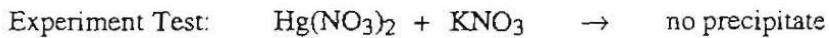
and thus the possible combinations are  $\text{Hg}(\text{NO}_3)_2$ ,  $\text{KI}$ ,  $\text{HgI}_2$ ,  $\text{KNO}_3$

Recognize that the product cannot be the reactant. Therefore the only possible products are



***Experimental Tests and Observations:***

The above test results indicate that  $\text{Hg}^{2+}_{(\text{aq})}$  must be present for the precipitate to form.



The test results indicate that  $\text{I}^{-}_{(\text{aq})}$  must be present for the precipitate to form..

***Additional Information and Conclusion:***

The CRC Handbook indicates that  $\text{HgI}_2$  is orange-red.  $\text{K}^+$  and  $\text{NO}_3^-$  ions are both colorless. If the precipitate is  $\text{KNO}_3$  it should be white rather than orange-red. The precipitate must be  $\text{HgI}_2$  and therefore the reaction must be,  $\text{Hg}(\text{NO}_3)_2 + 2\text{KI} \rightarrow \text{HgI}_{2(\text{s})} + 2\text{KNO}_3$



# APPENDIX (A)

## VC211 LABORATORY IMPORTANT INSTRUCTIONS

### PART I: GENERAL INSTRUCTIONS TO TA-s

**"Read very carefully all pages of this document and refer to the most recent information provided at the first few pages of the manual under Emergency Contact Information, Safety Announcement, and as provided by JI Canvas online. You must be familiar with all the information and be able to instruct the students on all the relevant information they need to complete the laboratory experience safely & successfully. This Appendix is subject to change by instructor and you may find some inconsistencies with common practice. Please be careful and work diligently and safely"**

**"With all my best wishes for a very safe and very successful laboratory experience"**

1. VC211 CANVAS Information: All TA's must be familiar with the content of the entire CANVAS site relation to VC211 as soon as possible and before meeting the students during the second week of classes. Please read all information in preparation to the lab and in particular pay special attention to the safety instructions.
2. Introduce Yourself to Students. Provide some background about yourself. Provide: Office location, office phone, email address, office hours.
3. Overview of the Day. Let the students know this session will take 2-3 hours. Outline briefly the activities for the day. Have them relax. Please make sure you arrive at least 5 minutes before start of your section schedule and direct students to the correct rooms for pre-lecture and then to the Lab area.
4. Take attendance. Circulate a sheet (lined paper with numbered column) and have each student sign in. Have everyone sign in regardless of whether they are officially enrolled in your lab section or not. Explain to the students not officially enrolled in the section that their presence here today does not guarantee them a spot in the lab for the rest of the semester and that only JI undergraduate office can add them into the course and/or lab/recitation section.
5. Laboratory Safety Inspection. Afterwards walk around the room and point out the safety items in the lab (e.g. fire extinguishers, fire blanket, safety shower, First Aid kit, phone for emergency calls). Explain to them that should a serious emergency arise, SJTU Campus Safety and SJTU emergency medical services must be notified immediately to handle the transport to the nearest medical service. Also, notify the chemistry department.
6. Conduct in lab. Talk about: 1) need for approved safety goggles, 2) proper attire (e.g. no scandals), 3) no food or drink in the lab, 4) proper handling of chemicals and disposable gloves are always available. Add that safety goggles can be stored in a locked drawer in the lab so that they don't have to remember to bring them with them each week. They should write their name on the strap of the goggles if they chose this option.

7. Clean up. Indicate to students that you will assign them clean up tasks before they leave. Before dismissing the students, each student is responsible for general cleaning of their bench work area (including cleaning the glassware). Then 2groups/week will alternate on a weekly basis to do more thorough cleaning (sink, floor, etc.) of the entire laboratory immediately before leaving the laboratory.
8. Groups. Undergraduate office will supply you with a class list and you will also assign students to a group. The entire class is divided into **9 class sections** and a TA is assigned to lead each section. Each section will be divided into groups of **4 students per group**. Each group conducts experiments jointly or individually as instructed. Each class section assembles in the laboratory area in the Chemistry Building B, second floor, according to the schedule posted on CANVAS and as shown by the class Syllabus.
9. Lab Reports (for details see later sections & course syllabus). Each student will be required to turn in an individual after-lab report (ALR) that includes pre-lab reports (PLE completed and PLQ ready to complete during lab session) at the start of each new experiment. The ALR will have missing data information, data analysis, discussions, conclusions & recommendations with references. Those missing sections are to be added to the ALR and submit at the start of the following week lab session. The team report form at the end of the experiment is merely a fill-in the blank outline and does not give all the questions you need to answer. You must follow the grading criteria for the reports and for the presentations (PPT) grading criteria posted on CANVAS and near the end of this appendix.

**"Grader must mark the report with grading comments & summarize score on front page. In matter of fact, grading comments should be notes of critical feedback to the student, so he/she can be well informed of improvements required to get better grade and or to be encouraged to be better next time. A mere grade only is insufficient grading efforts. The worse you can do if you give reports, homework or tests back to students without grades without a valid reason written on the returned work. Also, all permitted graded work must be returned to students in a reasonable timely fashion. Be very consistent and fair in grading legitimate work, regardless of your personal knowledge of the student. Your grading will affect the future academic standard of the student"**

For maximum points on the after-lab report, students need to respond to all directives and questions within the experiment itself and include specific examples from the data to support your conclusions. Tell students to check CANVAS for report formats and guidelines for the report contents and data analysis. Further guidelines are also posted on CANVAS on how to prepare PPT presentation on select experiments and writing a technical report that will help you prepare the experimental design report on Ca containing products. Also the class Syllabus has a table with specific instructions and due dates of reports and assignments and also posted on CANVAS.

10. SI Units, Conversion Factors, Scientific Notation, Dimensional Analysis Materials. Students should refer to the previous VC210 chemistry class notes on conversion of units and of handling the significant figures and scientific notations calculations correctly. I also, posted hints on significant figures on CANVAS along with many useful references and chemical data (including properties) that they may need in calculations.
11. Review. Using the above materials, you should review with the students the aforementioned topics on the blackboard and overhead projector.

12. Preview Next Week's Lab. All TA's are required to attend every week ahead (1 week ahead of each experiment) pre-lab orientation offered by the chemistry department. Please refer to the course syllabus and to CANVAS announcement for schedule of orientations and locations. Remind the students that they will need their safety goggles the second week that will be given by the chemistry department.
13. Laboratory Orientation Lecture: I will give about 1 ½ hours orientation lecture during the first week of classes to all students. Check CANVAS and with the undergraduate office for time, date and location of this only lecture. I expect TA's to attend the orientation to help me supervise the large number of students. I will cover most of the course requirements, assignments, and safety orientations posted on CANVAS.
14. The lab should be left neat and clean when you and your students leave. It is your responsibility to see that this happens (and not Linda Grimm's or her staff's responsibility). Suggestion: Give each group a cleanup assignment. Tell them they can't leave until you have checked off on this assignment. If a group fails to comply deduct professional points from the student's total. Observation: At the end of the lab period, students have the habit of sneaking out when your back is turned. Therefore, you need to lay down the law early in the semester on how cleanup will proceed. Typically, the last ½ hour of lab session should be enforced for cleanup.
15. If chemicals/reagents are in short supply or equipment or glassware is missing inform the Chemistry department immediately. If you teach an evening lab the support staff may be will not be there the entire evening. As regards glassware, keep in mind that most common items are available in the lab room – check the drawers under the lab benches. Check with the Chemistry department for the lab supply & inventory location.
16. When working with chemicals students and instructors are required to wear approved safety goggles and obtained from the Chemistry department. I expect you and your students not to violate this policy.
17. It is my expectation that you, the instructors, are knowledgeable and comfortable with each lab exercise before you walk into lab to oversee your students perform it. Do not go to the Chemistry department or staff for assistance or clarification during the lab. You must seek help before your lab session because the Chemistry department may be out of reach or busy with their work – they don't supervise the labs – that's your responsibility. I don't want to hear from your students "That's Dr.\_\_\_\_ didn't know what he was doing!" Having said that, I acknowledge that some of you have not done enough chemistry experiments before. Suggestion: The best way to familiarize yourself with a new lab exercise is to do it in advance yourself. This will be possible one week ahead of your lab session during lab orientation discussed in the previous paragraphs 13. Communicate with each other about lab problems/suggestions/ schedule. Leave notes on the TA's desk or blackboard for the next TA session. You might also want to visit a lab section that precedes yours and look in on the students performing the lab.
18. I invite (and encourage) you to contact me whenever you have a concern or question. It can be with how the course is running or to get clarification about an issue/assignment. I also encourage you to contact me regarding any student that is giving you problems or is experiencing problems (doesn't get along with her/his follow

group members). Please report to me students who are absent from recitation or lab more than once.

19. Do NOT add students to or drop students from your lab/rec section. Do not accept late assignments. On both these counts, send the student to me or JI undergraduate office.
20. Please establish office hours, be available during the posted time and let students know during the first week of classes and post schedule on CANVAS.
21. TA's must archive representative samples PDF scanned copies of all students' lab work and course materials (homework, tests, lab reports, project assignments reports & PPT). I must have archived copy first then the undergraduate office. The sample should be representatives of top, middle and low grades (2 samples each) for the entire class and not for each section.
22. If you know you can't make your assigned recitation/laboratory class sometime this semester please let me know in advance or if a situation arises where you can't make a class, please contact me and the lead TA as soon as possible. See course syllabus for contact information.
23. Do not make any decision of post announcements to students that they violate the instructions above and the instructions in the course syllabus. Also, consult with me and other TA-s before making any rational decisions. Announcements on CANVAS should be carefully posted after informing the lead TA-s and the lab instructor.
24. The students must adhere strictly to all the rules and regulations with respect to safety, health, and environmental protection as posted in this manual, or as posted everywhere else and as regulated by the governing laws of the land. In particular, smoking, drinking and eating are absolutely prohibited inside the laboratories. Proper safe clothing must be worn all the time before you can enter the labs. Students violating safety rules and regulations may be dismissed from the lab and their grades may be severely affected. Also, some violations may be prohibited by laws.

# **VC211 LABORATORY IMPORTANT INSTRUCTIONS**

## **PART II: LABORATORY SAFETY INSTRUCTIONS**

1. All Chemistry labs are set up once a week. There is no lab assistant assigned to clean up the lab after each section. Each instructor is responsible for every lab station, reagents, hoods and waste.

**Each instructor must:**

- Insist that every lab station is left clean
- Not store student lab products in lab drawers
- All glassware is emptied and rinsed
- All equipment is turned off
- Reagents and waste bottles are capped & labeled
- Balances are cleaned and turned off
- All Hoods:
  - Beaker/Burets must be emptied of solutions
  - Spill free
  - All waste is disposed of properly

Report any solution shortages or errors to the Dispensary Staff as soon as possible!

2. **Emergency contact numbers are shown by the course syllabus, on CANVAS, and in the emergency contact sheet at the beginning of the lab manual. You must ensure your safety, the safety of the students & others.** You must seek help if you face any safety issue so please make sure you have in your possession readily available emergency contact information.
3. Obtain Goggles and lab coats for students from the chemistry department. Make sure students return them back to their original place. Everyone in the lab must wear their Goggles and lab coats all time.
4. Only **Z87 Chemical Splash Resistant Goggles** may be worn in lab. It is the TA's **responsibility** to ensure that the correct types of goggles are worn. (No perforations, no colored lenses, no personal lab glasses and no safety glasses!)
5. Laboratory safety policy: **The following not permitted in lab: contact lenses, open toed shoes, sandals, shorts, food, or drinks. No smoking permitted.**
6. The dispensary staff for the laboratories is available by the Chemistry department to handle emergency situations. However, if you realize that your section or the one following it, will run out of solution or you need to report a piece of equipment that is inoperable, don't hesitate to call the Chemistry Department for help. Contact information provided by the course syllabus.
7. No students are permitted in lab **without** the Instructor or TA. In case of an emergency, requiring the Instructor to leave lab, the Chemistry department

should be contacted first to cover for a short time. **Lab must always be supervised! All lab doors are to be locked when not in use.**

8. All waste containers must be capped after lab!!
9. All the equipment and almost all of the chemicals needed are located inside the laboratory. Check with the chemistry department to see if there is an inventory log book in each room located in the “locked” instructor drawer with room diagrams and an alphabetical Listing/location.
  - Remember, hazardous substances are used in lab. This requires preplanning to respond safely to chemical spills. The cleanup of major or minor chemical spills should be done by the Instructor or experienced personnel. All minor spills must be cleaned up during the lab period! **All major spills must be reported IMMEDIATELY to the Chemistry department and to SJTU campus public safety.** The contact information listed in the course syllabus and in the emergency contact sheet at the beginning of the lab manual.
10. If an **accident** occurs during lab, the TA must determine the level of support necessary to help the injured student: **Emergency contact numbers are shown by the course syllabus, on CANVAS, and in the emergency contact sheet at the beginning of the lab manual.**
  - The Chemistry department is responsible for the laboratory management and can be contacted at the contact information listed by the course syllabus and by the emergency contact sheet in the beginning of the manual.
  - SJTU Campus Safety is available for all first aid assistance by calling 5474911.
  - **SJTU campus emergency medical services (tel: 54742400).**
  - The instructor is responsible for cleaning up the injured student’s area and maintaining a safe environment for the rest of the class.
  - All accidents must be reported to the Chemistry department and to SJTU public safety. All documentation must be on file within **24 hrs.** of the accident (see the attached form).
12. Check with the chemistry department to see if there are “Spill Clothes” for any lab accident victim in chemistry. Check the clothes location within the chemistry building that may have sweat shirts and sweat pants in various sizes. The only requirement is that the borrowed clothes are returned to the Chemistry Dispensary the next time the person is on campus.

## CHEMICAL HYGIENE PLAN

### EMERGENCY PROCEDURES

#### NOTIFICATION

In the event of an emergency or disaster, the SJTU Campus Safety should have primary responsibility for immediate response and shall-cooperate and coordinate with official emergency response authorities and University administration in accordance with established policies and procedures. SJTU Campus Safety, or alternate, will function as the Incident Commander.

#### CHEMICAL SPILLS

Hazardous substances used in laboratories require preplanning to respond safely to chemical spills. Knowledgeable and experienced personnel should only do the cleanup of chemical spills. Spill kits with instructions, absorbents, reactants, and personal protective equipment should be available to clean up incidental spills. An **incidental chemical spill** is one the laboratory staff is capable of handling safely without assistance from Campus Safety and/or emergency personnel. All other chemical spills are considered to be an **emergency release**.

##### Guidelines for Incidental Chemical Spills

- Alert people in the immediate area of the spill.
- Secure the area.
- If possible, try to ventilate the area by opening the fume hood sash and/or the windows. Fume hoods equipped with emergency buttons or high/low switches should be activated to increase ventilation in the area.
- Remove all sources of heat or ignition, if it can be done safely.
- **DO NOT** walk in the spill, this could spread it to other areas.
- Wear appropriate personal protective equipment, including safety goggles, gloves, and long-sleeve lab coats.
- Avoid breathing vapors from the spill. Avoid smoking, drinking, or eating.
- Confine the spill to a small area by surrounding it with a dike of absorbent material from the appropriate spill kit, working the outside of the spill area to the center.
- For inorganic acids and bases, use the appropriate spill kit to absorb and neutralize. Sodium bicarbonate is usually satisfactory for **dilute** solutions. Collect residue and place it in a waste container. Label as "hazardous waste" and notify the Manager of Laboratories for proper disposal. Caution: label on waste container may differ, depending on SJTU Chemistry Department labelling.
- For solvents, absorb the spill with a "Solvent Spill kit".
- For other chemicals, use appropriate spill kits or absorb spill with vermiculite. Label as "hazardous waste" and notify the Manager of Laboratories for proper disposal.
- Clean the spill area.
- **Report** the spill to the laboratory instructor, supervisor, or Manager of Laboratories or designee. See contact information (page ii of the first few pages of this lab manual), follow the additional safety instructions on page iii and this Appendix A.

### **Guidelines for Chemical Spill on the Body**

- Remove contaminated clothing at once and flood the exposed area with running water from a safety shower for at least 15 minutes.
- Make sure the chemical has not accumulated in your shoes.
- **Call SJTU campus emergency medical services ([tel:54742400](#))** to obtain immediate medical attention.
- **Report** the incident to the laboratory instructor, supervisor, or Manager of Laboratories or designee.

### **Guidelines for Chemical Splash in the Eye(s)**

- Immediately rinse the eye(s) and inner surface of the eyelid(s) with water continuously for at least 15 minutes. (Check for contact lenses).
- Hold the eye(s) open to ensure effective washing behind the eyelid(s). In the event glass or other foreign objects entered the eye, do not rub the eye.
- **Call SJTU campus emergency medical services ([tel:54742400](#))** to obtain immediate medical attention.
- **Report** the incident to the laboratory instructor, supervisor, or Manager of Laboratories or designee.

### **Guidelines for an Emergency Release**

- Attend to injured or contaminated persons and remove them from exposure, if possible.
- Alert people in the laboratory to evacuate.
- If spilled material is flammable, turn off ignition and heat sources, if it can be done safely.
- Secure the affected area.
- If possible try to ventilate the area by either opening the fume hood sash or the windows. Fume hoods equipped with an emergency button or high/low switch should be activated to increase ventilation in the area.
- **All major spills must be reported IMMEDIATELY to the Chemistry department ([tel. 54742802](#)) and to SJTU campus public safety ([tel. 54749111](#)).**
- Have a person knowledgeable of the incident and laboratory assist emergency personnel.
- **Report** the spill to the laboratory instructor, supervisor, or Manager of Laboratories or designee.

# **STANDARD OPERATING PROCEDURE (SOP)**

## **LABORATORY HEALTH CONSIDERATIONS**

### **ANNOUNCEMENT**

Some substances used in instructional laboratories may carry health risks for certain students despite our best efforts to minimize those risks. For example, students who have chemical sensitivities or those who are pregnant may or should have special concerns. Rather than leaving the assessment of these risks to the individual instructors, disciplines should discuss possible risks in the courses it teaches.

If the discipline identifies a course or courses that has (have) such risk, the instructor should put the following into the course syllabus and announce to the students at the first meeting of the course:

“Substances used/handled during the term may carry health risks for certain students despite efforts to minimize any potential hazard. For example, certain students may have allergies related to some chemicals. If any student has particular health concerns, seek medical advice and decides whether to continue or drop the class.”

Any information shared between the student and instructor is to be confidential and available to University Official on an “as needed” basis only.

**It is recommended to ensure safety and health standards for yourself and others. You should seek immediate help if you face any safety or health hazard issue, so please make sure you have in your possession a readily available emergency contact information of the local police, fire department and the nearest qualified emergency health care facility.**

**SHANGHAI JIAO TONG UNIVERSITY**  
**RISK MANAGEMENT OFFICE**  
**INJURY REPORT FOR NON-EMPLOYEES**  
**NOT TO BE USED FOR INJURY TO AN EMPLOYEE**

**REPORT BY TELEPHONE TO SJTU CAMPUS PUBLIC SAFETY OFFICE ALL ACCIDENTS RESULTING IN SERIOUS BODILY INJURY.**

**PRINT OR TYPE THIS REPORT IN DUPLICATE AND FORWARD TO THE RISK MANAGEMENT OFFICE.**

**DO NOT DISCUSS THE ACCIDENT WITH ANYONE. REFER ALL INQUIRIES AND CORRESPONDENCE TO THE RISK MANAGEMENT OFFICE AT ONCE.**

<b>INJURED PERSON AND INJURIES</b>	NAME OF PERSON INJURED			AGE	MARRIED <input type="checkbox"/> SINGLE <input type="checkbox"/>	
	ADDRESS: STREET CITY STATE ZIPCODE			PHONE NUMBER		
	APPARENT PHYSICAL LIMITATIONS (DESCRIBE)					
	NATURE & EXTENT OF INJURIES					
	FIRST AID RENDERED YES <input type="checkbox"/> NO <input type="checkbox"/> BY WHOM		TAKEN TO HOSPITAL YES <input type="checkbox"/> NO <input type="checkbox"/> BY WHOM			NAME OF HOSPITAL
<b>TIME &amp; PLACE</b>	DATE HOUR A.M. P.M.	LOCATION (STREET, BLDG.NO., ROOM NO.) BE SPECIFIC				
<b>DESCRIPTION OF ACCIDENT</b>	FULL DESCRIPTION OF ACCIDENT (BE SPECIFIC)					
	FULL DESCRIPTION OF CAUSE (BE SPECIFIC)					
	WAS ACCIDENT CAUSED BY A U-M EMPLOYEE YES <input type="checkbox"/> NO <input type="checkbox"/> NAME OF EMPLOYEE					
<b>TYPE AND CONDITION OF PREMISES</b>	<b>TYPE PREMISES</b> CLASSROOM <input type="checkbox"/> STAIRWAY <input type="checkbox"/> LABORATORY <input type="checkbox"/> LOADING AREA <input type="checkbox"/> HALLWAY <input type="checkbox"/> STREET <input type="checkbox"/> LOBBY OR FOYER <input type="checkbox"/> SIDEWALK <input type="checkbox"/> CONSTRUCTION SITE <input type="checkbox"/> OTHER <input type="checkbox"/>		<b>LIGHTING</b> NATURAL DAYLIGHT <input type="checkbox"/> WET <input type="checkbox"/> DRY <input type="checkbox"/> ARTIFICIAL LIGHT <input type="checkbox"/> SNOWY <input type="checkbox"/> ICY <input type="checkbox"/> RAIN <input type="checkbox"/> DARK OR UNLIGHTED <input type="checkbox"/>	<b>CONDITIONS</b> SURFACE UNEVEN <input type="checkbox"/> SURFACE DEFECTIVE <input type="checkbox"/> OTHER <input type="checkbox"/>	<b>SAFEGUARDS</b> BANNISTER OR RAILING <input type="checkbox"/> BARRICADE <input type="checkbox"/> MACHINE GUARDS <input type="checkbox"/> OTHER <input type="checkbox"/>	
<b>WITNESSES (VERY IMPORTANT)</b>	NAMES:		ADDRESSES: (STREET, CITY & STATE) PHONE			
DATE REPORT _____		SIGNATURE _____				
PREVENTION RECOMMENDATIONS:		NAME AND TITLE _____				
		U-M ADDRESS _____ PHONE# _____				

**VC211 SJTU CHEMISTRY DEPARTMENT LABORATORY SAFETY DISCLOSURE FORM:  
EACH STUDENT MUST SIGN FORM BEFORE CONDUCTING ANY LABORATORY WORK.**

	上海交通大学化学化工学院	文件编号	S112
	化学实验课安全承诺书	颁布日期	2008年9月
		修订版本	V1.2.201509

为保障学生和实验室的安全，进行化学实验课学习的学生在进入实验室之前，须仔细阅读本承诺书，树立牢固的安全意识，签认认可，然后才能取得做实验的资格。

1. 初次进入化学实验室时自觉自愿接受安全教育，了解使用水、电、气以及化学试剂的基本知识，熟悉实验室及其周边的安全出口通道、安全装置的位置与使用方法（包括紧急冲淋器、洗眼器、灭火器、报警装置等）。
2. 实验开始前，根据所做实验的安全要求做必要和充分的准备，制订实验方案，在得到教师允许的情况下进入实验环节，严格遵守操作规程进行实验。
3. 进入实验室穿好防护工作服，实验操作时佩戴防护眼镜。实验时思想集中，按照实验步骤认真操作，未经允许不得随意改动实验操作前后次序。
4. 严格按照要求取用各种化学试剂，不随意混合试剂或将试剂倒入水槽，按规定回收或倒入指定废液缸；危险品按照学校有关规定进行管理和使用，不得随意将化学试剂带出实验室。
5. 不穿短裤、短袖衬衫、裙子、高跟鞋、拖鞋、凉鞋等进入实验室，谨防化学品溅洒和滑倒。做实验时应束起长发，谨防机械损伤。
6. 在实验室室内不使用手机等电子设备、不大声喧哗打闹，不吸烟、不饮食，并自觉维持实验室卫生。
7. 实验结束后收拾实验室，关电、关水、关气、关灯、关窗、关门后，再离开实验室。
8. 学生应持有在校学习期间的医疗保险和人身意外伤害保险。
9. 本承诺书作为学生进实验室进行实验课学习的安全准入凭据，有效期仅限于本实验课教学期间。
10. 本承诺书一式两份，由学生和指导教师分别保管。

本人认真阅读了以上条款，对化学实验的各项安全管理制度已经知晓，并同意履行。若因违背上述承诺或化学实验基本安全规则造成意外安全事故，本人愿意接受处罚，并承担相应的责任。

课程名称				
实验时间	全	学年第	学期	周星期
班 级				
学生签名				
签名时间	年 月 日			

# VC211 LABORATORY IMPORTANT INSTRUCTIONS

## PART III

### GRADING, REPORTS, & PPT INSTRUCTIONS

#### 1. OVERALL COURSE GRADING

<b>GRADING GUIDE:</b> (Grading criteria may change at the discretion of the Instructor/TA)	<b>MAX. POINTS</b>	<b>MAX. % GRADE</b>
<p><b>5 EXPERIMENTS 150 POINTS EACH AS FOLLOWING:</b></p> <p>a. <b>PLE: PRE-LAB EXERCISES &amp; Quiz 30 POINTS</b>  b. <b>PLQ: POST-LAB &amp; DATA SHEET 40 POINTS</b>  c. <b>EXPT'L OPERATION LAB WORK 50 POINTS</b>  <i>"TA gives grade at end of experiment"</i>  d. <b>ALR: AFTER-LAB REPORT 30 POINTS</b>  <i>(based on quality, discussion &amp; analysis)</i></p>	<b>750</b>	<b>75%</b>
<b>EXPERIMENTAL DESIGN REPORT: Relies on experiments E1-E5 analyzing calcium (Ca) in commercial products</b>	<b>50</b>	<b>5%</b>
<b>FINAL EXAM: CLOSED BOOKS &amp; NOTES</b>	<b>150</b>	<b>15%</b>
<b>FINAL PPT ON SELECT E1-E5 EXPERIMENT: Each group to present one assigned experiment. You must document your experimental work with few photos to include in your reports and presentation. (25pts individual + 25pts group)</b>	<b>50</b>	<b>5%</b>
<b>TOTAL</b>	<b>1000</b>	<b>100%</b>

#### 2. GRADING ORDER: If not shown then refer to recent information from instructor or TA-s

Each lab report will be divided into parts. One TA is in charge of one part's grading for all 12 sections to guarantee a uniform criteria of scores. TAs will gather once a week to perform the grading duty under the supervision of coordinator and instructor.

### **3. REPORTS GATHERING**

Each TA must gather the After-Lab Report (**ALR**) for the **previous experiment** and the raw Data Sheet (**DS**) for the **current experiment**. Then deliver them ASAP to the TA grader tabulated above, in particular for sections on Friday night. DS should have your comments, i.e. student (A) did not finish PLE before lab, student (M) came late to the lab, student (R) violated safety rules, student (T) violated JI honor code, student (Z) was disruptive to lab session, etc.

### **4. IMPORTANT NOTES TO GRADER(S) (For TA-s only)**

- a. Grading must be finished and returned to each of us before the next lab begins. Please try to be fair and consistent while grading. If you want to know more about how to grade efficiently as well as to avoid some unpleasant issues, you are welcomed to attend the workshop called “Grading Issues & How to Prevent” sponsored by JI-CLT. This will count as 1 hour training for your Basic Teaching Assistant Certificate. You may register for it by sending an e-mail to [umji-clt@sjtu.edu.cn](mailto:umji-clt@sjtu.edu.cn). Please go to CANVAS and see CLT announcements for more information.
- b. Scores must be shown to the students before giving the reports back. I have already made the gradebook for you on CANVAS. All you need is to download the template, fill in scores, and upload the file again. You may easily do this with the help of Excel. Moreover, you can ask the JI TA mentor directly while uploading the grades or attend the TA CANVAS Training Workshops offered by JI-CLT.
- c. You may want to keep the nine collected raw data sheets for each conducted lab session in case some students come to you and ask for several points back, especially in analysis part. The raw data sheets allow you to judge the scores in a compelling way.
- d. Please send a copy of the gradebook to the main TA mentor for VC211 once you finished uploading that on CANVAS (copy will be used as a backup in case CANVAS crashes). Also, keep the TA mentor informed if you want to make legitimate changes to scores.
- e. The detailed grading criteria are listed in the course syllabus and in this appendix. You must enter the grades on the sheet after collecting it from the other TA's. To help you do this then each TA must enter the student identification on the grade sheet for each experiment by downloading it from CANVAS under “**FILES / LABORATORY MANUAL**”, then print & submit that for each ALR that the TA collects for you, along with the ALR reports of the previous week and the current experiment Data Sheet (DS). **No late report or assignment or work will be accepted after the due dates.**

### **5. IMPORTANT NOTES TO TA's:**

- a. To help the grader record the grades each TA must enter the student identification on the grade sheet given later in this appendix, then print & submit that for each ALR that the TA collects for you, along with the ALR reports of the previous week and the current experiment Data Sheet (DS).

- b. Try to record the behavior (good/poor) of each student during the lab so that you may judge the performance easily.
- c. Please check Pre-lab exercise (PLE) and Post-lab report (PLQ) during the lab, and sign your name after checking. Also, record and double-check the raw data of the students and give the raw **datasheet** to the grader. Always keep a backup (maybe photos) for your paper work in order to avoid unpredictable issues.
- d. Don't be late for the lab and ask the students to clean the lab before leaving. Please tell the lab manager from the Chemistry Department before you leave and ask him/her to confirm that you are allowed to leave. If your assigned cleaning team failed to do the lab cleaning & inspection then we must do that before leaving the lab.
- e. Feel free to ask the instructor or anyone that knows the answer if you need help to resolve any confusion. Also, you are encouraged to give corrections to the lab manual directly to Dr. Ting Sun.

# **WEEKLY EXPERIMENT DISCUSSIONS, FINAL ASSIGNED EXPERIMENT PRESENTATION, & FINAL Ca-ANALYSIS REPORT GUIDELINES**

**"Grading criteria and below guidelines may change at the discretion of the TA & instructor"**

## **I. WEEKLY EXPERIMENT DISCUSSIONS (maximum 50 points: 25 points individual students discussion & 25 points team efforts discussion)**

- a. It is a team efforts and everyone should participate to discuss their previous week experiment. Each group gives the presentation to the TA only while the other teams are working on their experiments. The TA must keep an eye on the other groups simultaneously. There is no time for the groups to give presentation to the entire class.
- b. Every time one person per team should give an oral-report within 5 minutes.
- c. We have 4 oral-presentations in total (the 5th experiment doesn't need to be reported), and most teams have 4 people in one team. Hence, you MUST take turns to give presentation. Each one should give oral-report at least one time.
- d. You don't need to prepare PPT and there is no equipment for you to show PPT. You only required to prepare an outline of key points for your oral-reports. The outline must be handed in after the presentation. Outline can either be printed or hand-written. Please use A4 paper or the SJTU lab-report paper.
- e. It is similar to the <Discussion> part in Post-Lab Questions (PLQ) report. You can discuss any topic you like related to the experiment. However, there is a very important difference between them. Oral-report results are discussed by one team and will give just one report. For <Discussion> part in report, you can of course consult each other as a team, but you MUST write down your own report. If a TA finds reports that are duplicated, then he may submit them to Honor Council as an evidence for violating JI honor codes.

## **II. FINAL ASSIGNED EXPERIMENT PRESENTATION (Team efforts worth 50 points)**

During the last week before final exam, each team is required to give 10-15 minutes presentation on one of the select experiments (E1-E5) that the TA will designate randomly. Students' PPT should include some photos documenting their experimental work. See Syllabus & Appendix A in manual for further guidelines.

## **I. FINAL REPORT PROJECT: Calcium Analysis in Ca-products (team efforts maximum worth 50 points)**

**EXPERIMENTAL DESIGN ASSIGNMENT:** This year the students will investigate on their own and propose experimental design and analysis of Ca content in common commercial products such as food, consumable products, or pharmaceutical vitamins. The students will rely on their work experience with experiments E1 to E5. The report should include abstract, introduction, theory, experimental setup, procedure, what data to measure (in suppliers supplementary products), & expected results. This design report should be condensed & similar to the depicted

reports of any of their experiments and no more than 5 pages (or as posted by the instructions under “FILES” on Canvas).

In previous assignments many students emailed me ask questions about this chemical analysis assignment. I am glad to give you the hints below. But remember what you did on your special report at the end of the term of VC210 chemistry class. The report efforts should be similar.

1. You can make hypothesis on the cation contents of the products, then postulate how to analyze the **calcium content (PPM or mg/L)**, how to precipitate Ca (isolated from the other cations), design experiment, write procedure, attempt to do pre-lab, data analysis, post lab and a conclusion report. Do not limit your gained knowledge from the lab (in particular experiment 5). It is open ended design where you will write short no more than 5 pages) challenging report to the instructor.
2. Transmittal letter is meant to teach you how to write a letter attached as a cover page to the report where you communicate with me, tell me your efforts, agony, difficulties faced, challenges, solutions and cost analysis, then use your expertise knowledge in presenting compelling evidences so I can support your project and give you grade for this assignment. The letter should be short and precise and will have technical data results that will only be used to support your project (the letter should be no more than about 1/2 page and different than the abstract).
3. Due date for this assignment is at start of your session week when you also present PPT about your assigned experiment.
4. This document under CANVAS/FILES/FINAL REPORT & PPT is only a guide. Further information can be obtained from the syllabus, laboratory manual (Appendix A).

## **LAB REPORT INFORMATION AND TURN IN DIRECTIONS**

Detailed instructions to below sections are given by Appendix A in the lab manual. Students must underline and highlight titles & subtitles, i.e. "**Introduction**", "**Theory**", "**Procedure**", "**Pre-Lab Exercises (PLE)**", etc.

- A) You will be required to turn in for each experiment and **at the start of each experiment a handwritten & typed sections individual comprehensive report** known as **After-Lab Report (ALR)**, with the following sections of the report all completed as instructed below with only few clearly shown exceptions. TA inspects the ALR prior to the start of the experiment (see schedule in the next table):
1. **Cover page** with experiment title, date, your Chinese & Pinyin name, student ID, section number and telephone number. Must also include under your name the names of your teammates in Chinese & Pinyin.
  2. **Introduction, Experimental Background, Theory, & Experimental Procedure:** similar to the lab manual but use your own version as much as possible. This section of the report due upon entering the lab to conduct the same experiment. Give clear concise theory and procedure. Do not copy others' versions of ALR! That could be regarded as a violation of copy rights.
  3. **Pre-Lab Exercise (PLE):** answer and complete all sections including providing data tables, as instructed in the lab manual for the PLE prior to entering the lab session. **TA will check the PLE and you cannot start any lab work if PLE is not completed.**
  4. **Post-Lab Questions (PLQ):** Must be completed and checked by the TA at the conclusion of each experiment before dismissal of the lab session. Ahead of lab session prepare all relevant sections including providing data tables, as instructed in the lab manual for the PLQ.
  5. **Datasheet:** Report all the experimental results on the provided datasheet and have it checked by the TA before dismissal of the lab session. You must have correct record, reasonable result and keep tidy & safe. At this point, gather all above report sections and attach to the next section.
  6. **Discussion of results, analysis, conclusions and recommendations:** These sections must be completed at the start of the next week experiment except for the last E5 experiment it will be due the same day you conduct the experiment. ALR should include data processing, analysis of results, discussion & correlation to theory, conclusions & recommendations (also complete exercise questions).
  7. **After-Lab Report (ALR):** All above sections are to be submitted to the TA as a final ALR report for each experiment. This final ALR report for each experiment is due the following week prior to the start of the next experiment except E5 will be due immediately after completing the experiment (the same day).

- B) **Laboratory discussion, Final PPT on randomly selected experiment (TA assigns from E1-E5), and Final Report Experimental Design for Ca –analysis: See below table for requirements & due dates.**

For maximum points on the ALR, you need to respond to all directives and questions within the experiment itself and include specific examples from the data to support your conclusions. Check CANVAS for report formats and guidelines for the report contents and data analysis. Further guidelines are also posted on CANVAS on how to prepare PPT presentation on select experiments (E1-E5) and writing a technical report that will help you prepare the experimental design report on Ca containing products. Following is ALR chart & a table that has specific instructions and due dates of reports and assignments:

**NO LATE WORK WILL BE ACCEPTED AFTER THE DUE DATE**

<b>VC211 AFTER-LAB REPORTS</b>	
<b>MUST SUBMIT 1-8</b>	<b>NOTES BELOW:</b>
1. REPORT STRUCTURE BELOW	Handwritten & typed (see instructions below) on one sided paper, single lines. Each student hands in individual report for each experiment except follow E5 group report format. <b>Must highlight &amp; underline titles &amp; subtitles</b>
2. COVER PAGE	New page, due as you enter the lab: Title, Name, student ID, team number, date.
3. OBJECTIVES INTRODUCTION BACKGROUND THEORY	New page, due as you enter the lab, no more than 2 pages summarized from the lab manual for the experiment and must use your own words as much as possible.
4. EXPERIMENTAL SETUP & PROCEDURE	Continued after (3), due as you enter the lab, copy exactly as written in the lab manual for the experiment and make corrections as needed
5. PLE: PRE-LAB EXERCISES	New page, due as you enter the lab, copy exactly from lab manual but answer all questions and make corrections as needed.
6. PLQ: AFTER-LAB QUESTIONS & DATA SHEET (DS)	New page, copy from lab manual before entering the lab but answer questions after completing the experiment, the questions related to the experiment, make tables & calculations ahead of experiment to complete in lab. Record your data in this section and also in the Data Sheet available in the front desk. Have TA check PLQ & datasheet.
7. DATA PROCESSING & ANALYSIS, DISCUSSION, OBSERVATIONS, CONCLUSIONS & RECOMMENDATIONS	Typed and handwritten. Continued after (6), due the following week, include detailed calculations, charts, diagrams, table, analysis of data with direct reference, discussion & plausible questions to confusions and hard to give answer by yourself (it is good to give your own independent thoughts), your analysis & observations, then 2-4 sentences concluding the experiment: conclusions, what did you learn, your recommendations, etc., all determined by yourself.
8. REFERENCES USED INCLUDING INTERNET RESOURCES	
END ALR	GATHER ALL ABOVE AND SUBMIT TO TA AS SOON AS YOU ENTER THE LAB DUE THE FOLLOWING WEEK EXCEPT E5 DUE AT END THE EXPERIMENT

**NO LATE WORK, HOMEWORK, ASSIGNMENT, REPORT, PPT, EXPERIMENT WILL BE ACCEPTED AFTER THE POSTED DUE DATES**

<b>Summary of</b>	<b>When? As assigned</b>	<b>Group vs. Individually</b>	<b>Where</b>	<b>Comments</b>
<b>Cover page, Introd., Expt'l Background, Theory, Procedure &amp; PLE</b>	Due at start of new experiment	Individually.	Hand to TA as entering lab to be checked in lab then turn with ALR	Must do prior to entering the lab to be familiar with entire experiment
<b>Post-Lab Questions (PLQ) &amp; Data sheet</b>	Due upon completion of experiment or as announced	Individually	Hand to TA after completing experiment to be checked in lab then turn with ALR	- Don't forget to copy the summary of your collected data into the TA's datasheet before leaving lab
<b>After-Lab Report (ALR)</b>	The completed ALR due in 1 week at start of the next experiment or as announced by instructor	Must be written individually. Only E5 is written by the group	Hand to TA before the next experiment begins  Group E5 report – before leaving lab same day	- Don't forget to copy the data to TA before leaving lab - Analyze data and make conclusions! E5 due same day after conducting the experiment
<b>Discussion (Presentation+ Abstracts) 5 min/group</b>	Discussion of last week experiment	Group – One student leads the discussion per Group (alternating)	In lab before the next experiment begins. E5 same day after conducting the experiment.	Share previous lab experience with others (in lab) & may ask few questions. E5 discussion same day after experiment.
<b>Final PPT on randomly selected experiment by the TA (one of E1-E5)</b>	Given by each group after completion of all experiments, during the 8 <sup>th</sup> week or as assigned.	All Group members participate in PPT.	Hand in copy of PPT to TA immediately after presentation (before the final exam).	Follow CANVAS for PPT guidelines & instructions
<b>Final REPORT on Experimental Design of Ca – analysis</b>	Written report due upon completion of all experiments ( <b>last week of Lab</b> ).	Reports due Individually.	Hand to TA immediately before making the PPT presentation in the 8 <sup>th</sup> week.	Follow instructions in syllabus and CANVAS on report guidelines & instructions

## VC211 CHEMISTRY EXPERIMENTS GRADE SHEET

SECTION#: _____ TA: _____		DATE: ____ / ____ / ____	
EXPERIMENT#: _____ TITLE: _____			
LAB ROOM: _____ NOTES: _____			
<b>GRADING GUIDE</b> <b>5 EXPERIMENTS 150 POINTS EACH AS FOLLOWING:</b> a. PLE: PRE-LAB EXERCISES      30 POINTS b. PLQ: POST-LAB & DATA SHEET      40 POINTS c. EXPT'L OPERATION LAB WORK      50 POINTS "TA gives grade at end of experiment" d. ALR: AFTER-LAB REPORT      30 POINTS <b>QUALITY, DISCUSSION &amp; ANALYSIS</b> <b>EXPERIMENTAL DESIGN REPORT: RELIES ON EXPERIMENTS 1 &amp; 3 ANALYZING CALCIUM CO IN COMMERCIAL PRODUCTS</b> <b>FINAL EXAM: CLOSED BOOKS &amp; NOTES</b> <b>FINAL PPT ON SELECT E1-E5 EXPT.: EACH GROUP ONE EXPERIMENT TO PRESENT PPT. DOCUMENT EXPERIMENTAL WORK WITH FEW PHOTOS.</b> <b>TOTAL</b> 1000      100%			
No	Student ID	Name	
			<b>1. PLE: Pre-lab Exercises (30pt)</b>
1			Give clear concise theory and procedure. Provide data table. Answer questions.
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
32			
33			
34			
35			
36			
37			
38			
39			
40			
41			
42			
43			
44			
45			

**"Grading criteria may change at the discretion of the TA & instructor"**

## VC211: Grading rubrics of PPT & FINAL REPORT

**"Grading criteria may change at the discretion of the TA & instructor"**

Topic Number: \_\_\_\_; Group Number: \_\_\_\_; PPT Score: \_\_\_\_ Report Score \_\_\_\_ Total Score: \_\_\_\_

PPT ON SELECT EXPRT CIRCLE 1: E1 E2 E3 E4 E5

PPT CRITERION	50 POINTS MAXIMUM	SCORE
1. Content/Structure	<b>20 POINTS</b>	
	a. Focus on topic (focus on topic for most of time)	
	b. Completeness (included all the key points, concepts and theories concerning with the topic)	
	c. Accuracy (accurate definition, concepts, chemical equations and expressions; accurate pictures, diagrams and graphs accompanying the clear notes)	
	d. Intuitive and easily-understandable (powerful pictures, graphs, diagrams and videos and proper animations to highlight the key points, use specific supplement materials, such as samples or models to support your words, write understandable English to present your ideas )	
	e. Organization of slides (arrange slides in the highly logic order, logical connections in materials)	
2. Format	<b>10 POINTS</b>	
	a. Length (have enough pages to use the probable assigned time well (10-15 min/topic; four group-mates cooperate and deliver a presentation))	
	b. Font (neither too big or too small, legible)	
	c. Correct use of PowerPoint animation (encourage animations to emphasize the key points, but animations should not be too much, time-consuming or attract too much attention away from of the topic)	
	d. Using pictures, graphs, diagrams, tables and illustrations rightly, they are really helpful for the understanding of your concepts or ideas.	
3. Q/A section	<b>10 POINTS</b>	
	a. At least two suitable questions with proper difficulty, which can help your audiences to improve their understanding about the key points and concepts well.	
	b. Correct and clear answer to your questions.	
4. Misc	<b>10 POINTS</b>	
	a. Handed in the ppt draft on time	
	b. Names & photos of group numbers + Topic No. + Group No	

	c. Using English correctly (no spelling mistakes, few grammatical errors)	
	d. Writing the ANNOTATION of speech at the Remarks/Memo part under each slide. Annotation is the document part of your speech. They are the words you have to speak to explain the concepts and ideas written on slides.	
	e. All referenced materials used in slides which are not original (Indicate the names, authors and pages of pictures, graphs, diagrams and tables of a book or an article, the website address if it is an internet materials, search engines cannot be counted as references)	
	f. Comfortable background color and template	
	g. Highlight the key words.	
	h. Well-arranged materials on each slide.	
REPORT WRITING	5 pages maximum <b>50 POINTS MAXIMUM</b>	
Design Experiment on Ca-Analysis in Commercial products. SEE NEXT FEW PAGES FOR ADDITIONAL GUIDELINES	a. <b>(10POINTS)</b> Transmittal cover letter to describe: importance of topic, accurate strategic team work to address the topic, organizational structure, clear planning of work, scheduling the work, monitoring, supervising, your team efforts and active contributions of each team member, result of efforts.	
	b. <b>(40POINTS)</b> Technical writing: Abstract, introduction, theory, experimental & laboratory set up, testing theory, results & analysis, conclusions & recommendations, references. Extensive charts of figures, tables, appendices and references should appear at end of report and they are additional pages to the technical content	

**To grading instructor:** circle the number of points that matches PPT & final report grade, then add total score to place on front page: (Maximum PPT points = 25pts individual + 25pts group)

**"Grading criteria may change at the discretion of the TA & instructor"**

PPT	0 PTS	20 PTS	25 PTS	30 PTS	35 PTS	40 PTS	45 PTS	50 PTS
FINAL REPORT	0 PTS	20 PTS	30 PTS	30 PTS	35 PTS	40 PTS	45 PTS	50 PTS

**GRADER'S COMMENTS:**

<b>SEC. #:</b>		<b>PPT GRADING SHEET: MAX. 25PTS INDIVIDUAL + 25PTS GROUP</b> "Grading criteria may change at the discretion of the TA & instructor"						
<b>Group</b>	<b>Name</b>	<b>Name</b>		<b>Rand</b>	<b>Indiv.</b>	<b>Group</b>	<b>PPT</b>	
<b>No.</b>	<b>Chinese</b>	<b>Pinyin</b>	<b>ID</b>		<b>PTS</b>	<b>PTS</b>	<b>PTS</b>	

## **VC211 Calcium Analysis in Ca-products**

### **TECHNICAL REPORT WRITING**

**EXPERIMENTAL DESIGN ASSIGNMENT:** This year the students will investigate on their own and propose experimental design and analysis of Ca content in common commercial products such as food, consumable products, or pharmaceutical vitamins. The students will rely on their work experience with experiments E1 to E5. The report should include abstract, introduction, theory, experimental setup, procedure, what data to measure (in suppliers supplementary products), & expected results. This design report should be condensed & similar to the depicted reports of any of their experiments and no more than 5 pages.

In previous assignments many students emailed me ask questions about this chemical analysis assignment. I am glad to give you the hints below. But remember what you did on your special report at the end of the term of VC210 chemistry class. The report efforts should be similar.

1. You can make hypothesis on the cation contents of the products, then postulate how to analyze the **calcium content (PPM or mg/L)**, how to precipitate Ca (isolated from the other cations), design experiment, write procedure, attempt to do pre-lab, data analysis, post lab and a conclusion report. Do not limit your gained knowledge from the lab (in particular experiment 5). It is open ended design where you will write short no more than 5 pages) challenging report to the instructor.
2. The document under CANVAS resources "EXPERIMENTAL DESIGN ASSIGNMENT.pdf" is only a guidance (see above).
3. Transmittal letter is meant to teach you how to write a letter attached as a cover page to the report where you communicate with me, tell me your efforts, agony, difficulties faced, challenges, solutions and cost analysis, then use your expertise knowledge in presenting compelling evidences so I can support your project and give you grade for this assignment. The letter should be short and precise and will have technical data results that will only be used to support your project. (the letter should be no more than about 1/2 page and different than the abstract).
4. Due date for this assignment is at start of your session this week when you also present PPT about your assigned experiment.

**I. FOR REPORTS ON ENGINEERING PROJECTS GENERAL QUALITY CRITERIA (laboratory reports format may be different)**

1. TECHNICAL CONTENT: PROJECT ASSESSMENT-FEASIBILITY, DESIGN PARAMETERS & STRATEGY FOR SOLUTION.
2. PLAN OF WORK & TASKS (FRAME WORK FOR THE TABLE OF CONTENTS). PROJECT PHASES CHART.
3. ECONOMICS & FEASIBILITY ANALYSIS.
4. REPORT FORMAT & QUALITY.
5. PROJECT OPTIMIZATION.
6. COMPUTER APPLICATIONS, MODELING & SIMULATION.
7. QUALITATIVE & QUANTITATIVE ANALYSIS OF PROJECT: TABLES, FIGURES, FLOW-CHARTS & ILLUSTRATIONS.
8. DETAILED PROCESSES AND FINAL PROJECT DESIGN.
9. SAFETY & ENVIRONMENTAL APPLICATION.
10. PERT / CPM/ CMMI / ISO'S ETC. APPLICATION.
11. ANALYSIS, CONCLUSIONS & RECOMMENDATIONS.
12. REFERENCES, APPENDICES & SUPPORT MATERIAL.
13. PROGRESS REPORTS.
14. PPT & ORAL PRESENTATIONS / DEMONSTRATION OF ENGINEERING PROJECT MANAGEMENT & LEADERSHIP – FINAL GRADE TEAM EFFORTS.

**II. FOR R&D TOPICS: STAGES OF TOPIC:** abstract, introduction, theory, laboratory & experimental setup, experimental results, analysis of results, compare to theory, then conclusions & recommendations, references. (**laboratory reports format may be different**)

## **Special TA Coordinator/Mentor Duties**

1. Lead the TA training in the Lab one time per week ahead of each experiment under the **lab course instructor's supervision**. Coordinates the schedule so all the assigned TA-s must attend.
2. Responsible to create the CANVAS grading and post final exam and final grades with the course instructor supervision.
3. Print and distribute the experiments data sheets to all the TA-s so the student teams can record their data.
4. Prepare easy to read, the emergency contact information for the labs and post them on the doors of the lab. Help course instructor supervise laboratory safety by printing/posting safety guidelines on CANVAS. Also, make sure TA-s enforce safety in labs and they wear their lab quotes. Remind TAs to clearly inform students that they are responsible for their own safety then others.
5. Help instructor of the course make announcements and post course materials on CANVAS. Also, approve all other TA-s announcements before posting to students on CANVAS. Announcements related to grading policy and creating new course criteria must be approved by the course instructor first before publishing on Canvas.
6. Make sure TA-s grade reports according to the course instructor's instructions, fairly & uniformly, making remarks on the reports, record grades on the reports and post them on CANVAS.
7. Provide course instructor's communication instructions in Chinese to the lab technicians and to the chemistry department. Coordinate scheduled meetings and agendas with the TA-s
8. Attend each TAs experimental orientation typically before the start of each main experiment. Distributing lab coats and lab manuals to TAs and collect them at the end of the course before the final week.
9. Attend the instructor's lecture presentations on each experiment only once preferably during the first lab session of each experiment. Then make immediately weekly announcement on what things that the TAs & students should do to ensure safety, equipment integrity, balance accuracy with time efficiency, give warning to violations, discourage students from engaging in plagiarism and violation of JI honor code, makeup experiments not allowed, to be prompt, careful, avoid intentional mistake, remind students to be alert and make them also share responsibility to help report violations immediately to prevent hazards and to protect equipment and facilities in the labs, etc. (like any useful related item in this document). Then instructor may make corrections and additions to it and so the other TAs may also do the same. One announcement per experiment that can be improved by other TAs that already had experience with the experiment.

10. Make announcement for students to attend lab sessions 10-15 minutes before start time, students must complete experimental work 40 minutes before end of lab sessions, record their data on the TAs datasheet for, then cleanup their bench areas and areas assigned by TAs and help the others cleanup.
11. Announce that students cannot make-up any part of the experiment without direct authorization of the instructor or TA. This is due to the tight time schedule for each experiment and to reduce chemical waste. For example, if a student is asked to titrate 0.500 moles of a sample but mistakenly he prepared a much higher amount of sample then the student should obviously ignore that sample because it would require much more titrant solution to achieve the end point, and that is clearly a waste of chemicals and hazard to the environment. Students to report the actual data results and discuss thoroughly why they could not finish certain parts of the experiment, what mistakes they did, how did they learn from that, how did they balance accuracy with efficiency, and to discuss any other relevant issue (such as disregarding certain data because of this and that and statistics). If students intentionally make mistakes such as ignoring procedures and instructions then they should be warned and probably dismissed from lab if they cause serious danger or harm to the environment. The TAs may deduct points from the students' score of the experiment in faulty errors as they may deem appropriate. This policy is aimed to make sure students have enough time to complete the remains of the experiment to the best they can, protect the environment, and minimize chemical waste and disposal. Warn students about chemical disposal and safety violations.
12. Supervise constantly the weighing balance area in the Chemistry department near the labs during the second week when students conduct experiment E1. Ensures protection of equipment and glassware, give instructions on handling weighing balances, weighing vials, and proper procedure to weighing samples.
13. Remind TAs of their responsibilities and report to course instructor serious violations. Also, make sure that TAs do not dismiss students out of the lab session when they complete their experimental work ahead of others. Instead the TA should have those students help in cleaning the lab and assign them different duties that can help complete the lab session in a timely successful manner but not to help other students conducting their experiments. However they can help other students show them special skills and techniques (they know) on how to avoid experimental errors and use more common sense.
14. Ensures that TAs enforces the lab cleaning duties such as ensure that students clean their glassware, bench area, remove clutters, clean equipment and their area, wipe the sinks clean, sweep & mop floors, empty garbage cans, proper disposal of chemical waste, shutting down power lights (but not hoods power if lab requires ventilation and not to temper with emergency equipment but insure they are functional), then bring lab condition so it is properly ready for the next session and as instructed by the chemistry department.

## APPENDIX (B): ADDITIONAL REFERENCES

**Students are recommended to buy the following reference laboratory manual book as a valuable resource for information and in particular the introductory information and the appendices.**

**Nelson, Kemp, & Stoltzfus, ANNOTATED INSTRUCTOR EDITION-LABORATORY EXPERIMENTS, Chemistry the Central Science, 12<sup>th</sup> edition, Prentice Hall (Pearson Publisher), 2012 (ISBN-13: 978-0-321-71197-7 / ISBN-10: 0-321-71197-1, USA.**

**The following are the Contents of the book:**

ANNOTATED INSTRUCTOR'S EDITION		iv Contents
<h2 style="text-align: center;">Contents</h2>		
<b>In Memoriam</b>	vi	
<b>To the Instructor</b>	vii	
<b>General Guidelines for the Disposal of Laboratory Waste</b>	viii	
<b>Student Laboratory Desk Items</b>	x	
<b>Necessary Equipment and Materials and Helpful Hints</b>	xi	
1 Basic Laboratory Techniques (2.5 hr) <sup>*</sup> [1] <sup>**</sup>	1	
2 Identification of Substances by Physical Properties (2.5 hr) [1, 2]	19	
3 Separation of the Components of a Mixture (2.5 hr) [1, 2]	33	
4 Chemical Reactions (2.5 hr) [3, 4]	43	
5 Chemical Formulas (2.5 hr) [3, 4]	55	
6 Chemical Reactions of Copper and Percent Yield (2 hr) [3, 4]	69	
7 Chemicals in Everyday Life: What Are They and How Do We Know? (2 hr) [4, 13]	79	
8 Gravimetric Analysis of a Chloride Salt (3 hr) [3, 4, 13]	91	
9 Gravimetric Determination of Phosphorus in Plant Food (3 hr) [3, 4, 18]	103	
10 Paper Chromatography: Separation of Cations and Dyes (2.5 hr) [1, 4, 5]	115	
11 Molecular Geometries of Covalent Molecules: Lewis Structures and the VSEPR Model (2.5 hr) [8, 9]	127	
12 Atomic Spectra and Atomic Structure (2.5 hr) [6]	145	
13 Behavior of Gases: Molar Mass of a Vapor (3 hr) [10]	161	
14 Determination of R: The Gas Law Constant (2.5 hr) [10]	173	
		15 Activity Series (2.5 hr) [4, 20] 185
		16 Electrolysis, the Faraday, and Avogadro's Number (2.5 hr) [20] 195
		17 Electrochemical Cells and Thermodynamics (3 hr) [19, 20] 203
		18 The Chemistry of Oxygen: Basic and Acidic Oxides and the Periodic Table (2.5 hr) [16, 22] 221
		19 Colligative Properties: Freezing Point Depression and Molar Mass (2.5 hr) [13] 237
		20 Titration of Acids and Bases (3 hr) [4, 13, 17] 251
		21 Reactions in Aqueous Solutions: Metathesis Reactions and Net Ionic Equations (2.5 hr) [4, 13, 15, 17] 265
		22 Colorimetric Determination of an Equilibrium Constant in Aqueous Solution (2.5 hr) [15] 277
		23 Chemical Equilibrium: Le Chatelier's Principle (2.5 hr) [15] 293
		24 Hydrolysis of Salts and pH of Buffer Solutions (3 hr) [16, 17] 305
		25 Determination of the Dissociation Constant of a Weak Acid (3 hr) [16, 17] 325
		26 Titration Curves of Polyprotic Acids (3 hr) [17] 343
		27 Determination of the Solubility-Product Constant for a Sparingly Soluble Salt (3 hr) [15, 17] 361
		28 Heat of Neutralization (2.5 hr) [5, 19] 375
		29 Rates of Chemical Reactions I: A Clock Reaction (2.5 hr) [14] 389
		30 Rates of Chemical Reactions II: Rate and Order of $H_2O_2$ Decomposition (3 hr) [14] 407
		31 Introduction to Qualitative Analysis (3-6 hr) [15-17] 423
		32 Abbreviated Qualitative Analysis Scheme (12 hr) [15-17] 449
		Part I: Chemistry of Group 1 Cations: $Pb^{2+}$ , $Ag^+$ , $Hg_2^{2+}$ 452
		Part II: Chemistry of Group 2 Cations: $Pb^{2+}$ , $Cd^{2+}$ , $Bi^{3+}$ , $Sb^{3+}$ 457
		Part III: Chemistry of Group 3 Cations: $Tl^{+}$ , $Ni^{2+}$ , $Mn^{2+}$ , $Al^{3+}$ 465
		Part IV: Chemistry of Group 4 Cations: $Ba^{2+}$ , $Ca^{2+}$ , $NH_4^+$ , $Na^+$ 473
		Part V: Chemistry of Anions: $SO_4^{2-}$ , $NO_3^-$ , $CO_3^{2-}$ , $Cl^-$ , $Br^-$ , $I^-$ , $CrO_4^{2-}$ , $PO_4^{3-}$ , $S^{2-}$ , $SO_3^{2-}$ 479
		Part VI: Analysis of a Simple Salt 491
		33 Colorimetric Determination of Iron (3 hr) [23] 509
		34 Solubility and Thermodynamics (3 hr) [15, 17, 20] 523
		35 Analysis of Water for Dissolved Oxygen (2.5 hr) [18] 533
		36 Preparation and Reactions of Coordination Compounds: Oxalate Complexes (3 hr) [23] 547

<sup>\*</sup>Approximate time required to complete experiment.

<sup>\*\*</sup>The numbers in brackets after experiment titles refer to chapter(s) in the twelfth edition of *Chemistry: The Central Science* by Brown, LeMay, Bursten, Murphy and Woodward that are relevant to the experiment.

37	Oxidation-Reduction Titrations I: Determination of Oxalate (3 hr) [20, 23]	559
38	Preparation of Sodium Bicarbonate and Sodium Carbonate (3 hr) [3, 4]	569
39	Oxidation-Reduction Titrations II: Analysis of Bleach (3 hr) [20, 23]	577
40	Molecular Geometry: Experience with Models (2 hr) [9]	589
41	Preparation of Aspirin and Oil of Wintergreen (2.5 hr) [25]	603
42	Analysis of Aspirin (3 hr) [17, 25]	611
43	Crystalline Solids (2.5 hr) [12]  Appendices	621 A1
	A The Laboratory Notebook	A3
	B Chemical Arithmetic	B8
	C Graphical Interpretation of Data: Calibration Curves and Least-Squares Analysis	C17
	D Summary of Solubility Properties of Ions and Solids	D21
	E Solubility-Product Constants for Compounds at 25°C	E23
	F Dissociation Constants for Acids at 25°C	F25
	G Dissociation Constants for Bases at 25°C	G27
	H Selected Standard Reduction Potentials at 25°C	H28
	I Spreadsheets	I31
	J Qualitative-Analysis Techniques	J38
	K Answers to Selected Pre-lab Questions	K40
	L Vapor Pressure of Water at Various Temperatures	L66
	M Names, Formulas, and Charges of Common Ions	M67
	N Some Molar Masses	N69
	O Basic SI Units, Some Derived SI Units, and Conversion Factors	O71
	P Composition of Commercial Reagent Acids and Bases	P73
	Q Additional Answers to Pre Lab Questions for Instructors	Q74

**Periodic Table of the Elements**

**Modified by Dr. Hammade**

Main Group Representative Elements		Main Group Representative Elements																			
$1s^1$ H	$2s^2$ Be													$3s^2$ Al	$4s^2$ Si	$5s^2$ P	$6s^2$ S	$7s^2$ Cl	$8s^2$ F		
1.00794	9.012182													10.811	12.0107	14.00667	15.9994	18.998403	20.1797		
1 $A^1$	2 $A^2$															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
1 $A^1$	2 $A^2$															1.3	1.4	1.5	1.6	1.7	1.8
2 $B^3$	3 $B^4$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
2 Li	3 Na															$3p^1$ Al	$4p^1$ Si	$5p^1$ P	$6p^1$ S	$7p^1$ Cl	$8p^2$ F
6.941	22.989770															1.3	1.4	1.5	1.6	1.7	1.8
3 $C^5$	4 $C^6$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
3 $Li^1$	4 $Na^2$															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
12	12															1.3	1.4	1.5	1.6	1.7	1.8
3 $Mg^3$	4 $Mg^4$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
11	12															$3p^1$ Al	$4p^1$ Si	$5p^1$ P	$6p^1$ S	$7p^1$ Cl	$8p^2$ F
30.0983	40.078															1.3	1.4	1.5	1.6	1.7	1.8
3 $Sc^{21}$	4 $Sc^{22}$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
4 $K^{19}$	5 $K^{20}$															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
39.0983	44.955910															1.3	1.4	1.5	1.6	1.7	1.8
4 $Ca^{21}$	5 $Ca^{22}$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
39.0983	44.955910															$3p^1$ Al	$4p^1$ Si	$5p^1$ P	$6p^1$ S	$7p^1$ Cl	$8p^2$ F
4 $Sc^{21}$	5 $Sc^{22}$															1.3	1.4	1.5	1.6	1.7	1.8
38	39															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
5 $Rb^{37}$	6 $Rb^{39}$															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
88.4678	87.62															1.3	1.4	1.5	1.6	1.7	1.8
5 $Sc^{37}$	6 $Sc^{39}$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
56	57															$3p^1$ Al	$4p^1$ Si	$5p^1$ P	$6p^1$ S	$7p^1$ Cl	$8p^2$ F
132.90545	137.327															1.3	1.4	1.5	1.6	1.7	1.8
6 $Cs^{55}$	7 $Cs^{57}$															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
122.02	122.03															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
6 $Lu^{55}$	7 $Lu^{57}$															1.3	1.4	1.5	1.6	1.7	1.8
174.967	178.49															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
7 $Fr^{87}$	88															$3p^1$ B	$4p^1$ C	$5p^1$ N	$6p^1$ O	$7p^1$ F	$8p^2$ Ne
122.11	122.11															1.3	1.4	1.5	1.6	1.7	1.8
7 $Fr^{87}$	88															10.811	12.0107	14.00667	15.9994	18.998403	20.1797
122.02	122.03															$3p^1$ Al	$4p^1$ Si	$5p^1$ P	$6p^1$ S	$7p^1$ Cl	$8p^2$ F
122.02	122.03															1.3	1.4	1.5	1.6	1.7	1.8
INNER TRANSITION ELEMENTS																					
Lanthanide series		$4f^{57}$ La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu	72 At				
		138.9055	140.90765	144.24	144.24	140.90765	145	150.36	151.964	157.25	158.92534	162.50	164.93032	167.259	168.94241	173.04	173.04	ATOMIC WT.			
Actinide series		$5f^{89}$ Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	102 No	102 No	ATOMIC WT.			
		122.03	232.0381	231.03588	238.02891	237.051	244.061	243.06	247.071	251.081	252.081	257.101	258.101	259.101	259.101	259.101	ATOMIC WT.				

<sup>a</sup> The labels on top (1A, 2A, etc.) are common American usage. The labels below these (1, 2, etc.) are those recommended by the International Union of Pure and Applied Chemistry (IUPAC).

The names and symbols for elements 113 and above have not yet been decided.

Atomic weights in brackets are the names of the longest-lived or most important isotope of radioactive elements.

Further information is available at <http://www.webelements.com>

\*\* Discovered in 2010, element 117 is currently under review by IUPAC.

**University of Michigan – Shanghai Jiao Tong University (UM-SJTU JI)**

**800 Dongchuan Road, Shanghai, China**

**200240 Email: [ting.sun@sjtu.edu.cn](mailto:ting.sun@sjtu.edu.cn)**

**Web: <http://umji.sjtu.edu.cn>**