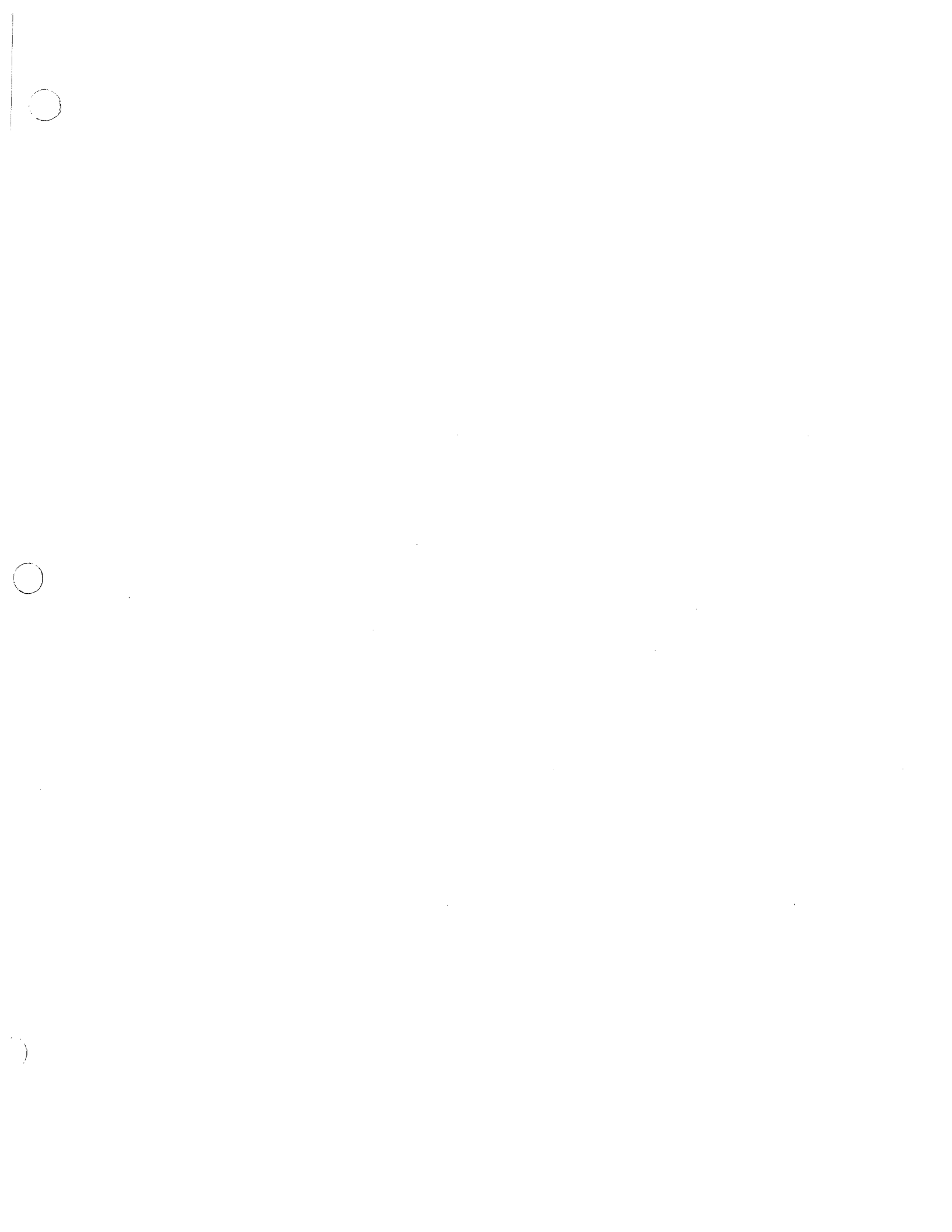


Magnet Analytical Chemistry
Laboratory Manual
2015

Name: _____



INFORMATION ACKNOWLEDGEMENT

I have received, read and understand the class expectations and lab safety contract for Mrs. Zado's chemistry class. (Signatures from both parents/guardians requested, if possible. Thank you.)

Student Name (last, first middle) Printed

Block

Grade

Student Signature

Home Phone Number

Parent/Guardian Signature

Daytime Phone Number

Parent/Guardian Signature

Daytime Phone Number

Daytime E-mail address (1)

E-mail address (2)

STUDENT INFORMATION

Nicknames: _____

DOB: _____

Are you in a regularly scheduled after school activity? (sports, drama, band, job, etc.)

Do you have any physical problems that I should be aware of? If yes, please explain.

What was your last math class? _____ Grade earned: _____

How did you do in biology? _____ Grade earned: _____

Do you have access to a computer, printer and internet at home? _____

E-Mail address: _____

On the back please tell me something about yourself. Please write in proper English. Include something about yourself, your family, home life, your goals and ambitions. I am interested in learning about your hobbies, interests and anything else which would help me to know you better.

Thank you!

Mrs. Zado

Flinn Scientific's Student Safety Contract

PURPOSE

Science is a hands-on laboratory class. You will be doing many laboratory activities which require the use of hazardous chemicals. Safety in the science classroom is the #1 priority for students, teachers, and parents. To ensure a safe science classroom, a list of rules has been developed and provided to you in this student safety contract. These rules must be followed at all times. Two copies of the contract are provided. One copy must be signed by both you and a parent or guardian before you can participate in the laboratory. The second copy is to be kept in your science notebook as a constant reminder of the safety rules.

GENERAL RULES

1. Conduct yourself in a responsible manner at all times in the laboratory.
2. Follow all written and verbal instructions carefully. If you do not understand a direction or part of a procedure, ask the instructor before proceeding.
3. Never work alone. No student may work in the laboratory without an instructor present.
4. When first entering a science room, do not touch any equipment, chemicals, or other materials in the laboratory area until you are instructed to do so.
5. Do not eat food, drink beverages, or chew gum in the laboratory. Do not use laboratory glassware as containers for food or beverages.
6. Perform only those experiments authorized by the instructor. Never do anything in the laboratory that is not called for in the laboratory procedures or by your instructor. Carefully follow all instructions, both written and oral. Unauthorized experiments are prohibited.
7. Be prepared for your work in the laboratory. Read all procedures thoroughly before entering the laboratory.
8. Never fool around in the laboratory. Horseplay, practical jokes, and pranks are dangerous and prohibited.
9. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Bring only your laboratory instructions, worksheets, and/or reports to the work area. Other materials (books, purses, backpacks, etc.) should be stored in the classroom area.
10. Keep aisles clear. Push your chair under the desk when not in use.

11. Know the locations and operating procedures of all safety equipment including the first aid kit, eyewash station, safety shower, fire extinguisher, and fire blanket. Know where the fire alarm and the exits are located.
12. Always work in a well-ventilated area. Use the fume hood when working with volatile substances or poisonous vapors. Never place your head into the fume hood.
13. Be alert and proceed with caution at all times in the laboratory. Notify the instructor immediately of any unsafe conditions you observe.
14. Dispose of all chemical waste properly. Never mix chemicals in sink drains. Sinks are to be used only for water and those solutions designated by the instructor. Solid chemicals, metals, matches, filter paper, and all other insoluble materials are to be disposed of in the proper waste containers, not in the sink. Check the label of all waste containers twice before adding your chemical waste to the container.
15. Labels and equipment instructions must be read carefully before use. Set up and use the prescribed apparatus as directed in the laboratory instructions or by your instructor.
16. Keep hands away from face, eyes, mouth and body while using chemicals or preserved specimens. Wash your hands with soap and water after performing all experiments. Clean all work surfaces and apparatus at the end of the experiment. Return all equipment clean and in working order to the proper storage area.
17. Experiments must be personally monitored at all times. You will be assigned a laboratory station at which to work. Do not wander around the room, distract other students, or interfere with the laboratory experiments of others.
18. Students are never permitted in the science storage rooms or preparation areas unless given specific permission by their instructor.
19. Know what to do if there is a fire drill during a laboratory period; containers must be closed, gas valves turned off, fume hoods turned off, and any electrical equipment turned off.
20. Handle all living organisms used in a laboratory activity in a humane manner. Preserved biological materials are to be treated with respect and disposed of properly.

21. When using knives and other sharp instruments, always carry with tips and points pointing down and away. Always cut away from your body. Never try to catch falling sharp instruments. Grasp sharp instruments only by the handles.
22. If you have a medical condition (e.g., allergies, pregnancy, etc.), check with your physician prior to working in lab.

CLOTHING

23. Any time chemicals, heat, or glassware are used, students will wear laboratory goggles. There will be no exceptions to this rule!
24. Contact lenses should not be worn in the laboratory unless you have permission from your instructor.
25. Dress properly during a laboratory activity. Long hair, dangling jewelry, and loose or baggy clothing are a hazard in the laboratory. Long hair must be tied back and dangling jewelry and loose or baggy clothing must be secured. Shoes must completely cover the foot. No sandals allowed.
26. Lab aprons have been provided for your use and should be worn during laboratory activities.

ACCIDENTS AND INJURIES

27. Report any accident (spill, breakage, etc.) or injury (cut, burn, etc.) to the instructor immediately, no matter how trivial it may appear.
28. If you or your lab partner are hurt, immediately yell out "Code one, Code one" to get the instructor's attention.
29. If a chemical splashes in your eye(s) or on your skin, immediately flush with running water from the eyewash station or safety shower for at least 20 minutes. Notify the instructor immediately.
30. When mercury thermometers are broken, mercury must not be touched. Notify the instructor immediately.

HANDLING CHEMICALS

31. All chemicals in the laboratory are to be considered dangerous. Do not touch, taste, or smell any chemicals unless specifically instructed to do so. The proper technique for smelling chemical fumes will be demonstrated to you.
32. Check the label on chemical bottles twice before removing any of the contents. Take only as much chemical as you need.
33. Never return unused chemicals to their original containers.

FLINN SCIENTIFIC INC.

*"Your Safer Source
for Science Supplies"*

P.O. Box 219, Batavia, IL 60510
1-800-452-1261 • Fax: (866) 452-1436
flinn@flinnsci.com • www.flinnsci.com

Flinn Scientific's Student Safety Contract

34. Never use mouth suction to fill a pipet. Use a rubber bulb or pipet pump.
35. When transferring reagents from one container to another, hold the containers away from your body.
36. Acids must be handled with extreme care. You will be shown the proper method for diluting strong acids. Always add acid to water, swirl or stir the solution and be careful of the heat produced, particularly with sulfuric acid.
37. Handle flammable hazardous liquids over a pan to contain spills. Never dispense flammable liquids anywhere near an open flame or source of heat.
38. Never remove chemicals or other materials from the laboratory area.
39. Take great care when transporting acids and other chemicals from one part of the laboratory to another. Hold them securely and walk carefully.

HANDLING GLASSWARE AND EQUIPMENT

40. Carry glass tubing, especially long pieces, in a vertical position to minimize the likelihood of breakage and injury.
41. Never handle broken glass with your bare hands. Use a brush and dustpan to clean up broken glass. Place broken or waste glassware in the designated glass disposal container.
42. Inserting and removing glass tubing from rubber stoppers can be dangerous. Always lubricate glassware (tubing, thistle tubes, thermometers, etc.) before attempting to insert it in a stopper. Always protect your hands with towels or cotton gloves when inserting glass tubing into, or removing it from, a rubber stopper. If a piece of glassware becomes "frozen" in a stopper, take it to your instructor for removal.
43. Fill wash bottles only with distilled water and use only as intended, e.g., rinsing glassware and equipment, or adding water to a container.
44. When removing an electrical plug from its socket, grasp the plug, not the electrical cord. Hands must be completely dry before touching an electrical switch, plug, or outlet.
45. Examine glassware before each use. Never use chipped or cracked glassware. Never use dirty glassware.
46. Report damaged electrical equipment immediately. Look for things such as frayed cords, exposed wires, and loose

connections. Do not use damaged electrical equipment.

47. If you do not understand how to use a piece of equipment, ask the instructor for help.
48. Do not immerse hot glassware in cold water; it may shatter.

HEATING SUBSTANCES

49. Exercise extreme caution when using a gas burner. Take care that hair, clothing and hands are a safe distance from the flame at all times. Do not put any substance into the flame unless specifically instructed to do so. Never reach over an exposed flame. Light gas (or alcohol) burners only as instructed by the teacher.
50. Never leave a lit burner unattended. Never leave anything that is being heated or is visibly reacting unattended. Always turn the burner or hot plate off when not in use.
51. You will be instructed in the proper method of heating and boiling liquids in test tubes. Do not point the open end of a test tube being heated at yourself or anyone else.
52. Heated metals and glass remain very hot for a long time. They should be set aside to cool and picked up with caution. Use tongs or heat-protective gloves if necessary.
53. Never look into a container that is being heated.
54. Do not place hot apparatus directly on the laboratory desk. Always use an insulating pad. Allow plenty of time for hot apparatus to cool before touching it.
55. When bending glass, allow time for the glass to cool before further handling. Hot and cold glass have the same visual appearance. Determine if an object is hot by bringing the back of your hand close to it prior to grasping it.

QUESTIONS

56. Do you wear contact lenses?
 YES NO
57. Are you color blind?
 YES NO
58. Do you have allergies?
 YES NO
If so, list specific allergies _____

AGREEMENT

I, _____ (student's name) have read and agree to follow all of the safety rules set forth in this contract. I realize that I must obey these rules to ensure my own safety, and that of my fellow students and instructors. I will cooperate to the fullest extent with my instructor and fellow students to maintain a safe lab environment. I will also closely follow the oral and written instructions provided by the instructor. I am aware that any violation of this safety contract that results in unsafe conduct in the laboratory or misbehavior on my part, may result in being removed from the laboratory, detention, receiving a failing grade, and/or dismissal from the course.

Student Signature

Date

Dear Parent or Guardian:

We feel that you should be informed regarding the school's effort to create and maintain a safe science classroom/laboratory environment.

With the cooperation of the instructors, parents, and students, a safety instruction program can eliminate, prevent, and correct possible hazards.

You should be aware of the safety instructions your son/daughter will receive before engaging in any laboratory work. Please read the list of safety rules above. No student will be permitted to perform laboratory activities unless this contract is signed by both the student and parent/guardian and is on file with the teacher.

Your signature on this contract indicates that you have read this Student Safety Contract, are aware of the measures taken to ensure the safety of your son/daughter in the science laboratory, and will instruct your son/daughter to uphold his/her agreement to follow these rules and procedures in the laboratory.

Parent/Guardian Signature

Date

FLINN SCIENTIFIC INC.

*"Your Safer Source
for Science Supplies"*

P.O. Box 219, Batavia, IL 60510
1-800-452-1261 • Fax: (866) 452-1436
flinn@flinnsci.com • www.flinnsci.com

Periodic Table of the Elements

IA	IIA		IIIB - VIII										IB	IIB		IIIA	IVA	VA	VIA	VIIA	VIIIA																																																													
1	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																																															
H 1.008	He 4.003	Li 6.941	Be 9.012	B 10.81	C 12.01	N 14.01	O 16.00	F 19.00	Ne 20.18	Na 23.00	Mg 24.30	K 39.10	Ca 40.08	Sc 44.96	Ti 47.88	V 50.94	Cr 52.00	Mn 54.94	Fe 55.85	Co 58.93	Ni 58.69	Cu 63.55	Zn 65.38	Ga 69.72	Ge 72.59	As 74.92	Se 78.96	Br 79.90	Kr 83.80	Rb 85.47	Sr 87.62	Y 88.91	Zr 91.22	Nb 92.91	Mo 95.94	Tc (98)	Ru 101.1	Rh 102.9	Pd 106.4	Ag 107.9	Cd 112.4	In 114.8	Sn 118.7	Sb 121.8	Te 127.6	I 126.9	Xe 131.3	Cs 132.9	Ba 137.3	Lu 175.0	Hf 178.5	Ta 180.9	W 183.8	Re 186.2	Os 190.2	Pt 195.1	Au 197.0	Hg 200.6	Tl 204.4	Pb 207.2	Bi 209.0	Po (209)	At (210)	Rn (222)	Fr (223)	Ra 226.0	Lr (260)	Rf (261)	Db (262)	Sg (263)	Bh (262)	Hs (265)	Mt (266)	Ds (281)	Rg (280)	Cn (285)	Uut (284)	Ff (289)	Uup (288)	Lv (293)	Uus (294)	Uuo (294)
57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120																			
La 138.9	Ce 140.9	Pr 140.9	Nd 144.2	Pm (145)	Sm 150.4	Eu 152.0	Gd 157.2	Tb 158.9	Dy 162.5	Ho 164.9	Er 167.3	Tm 168.9	Yb 173.0	Lu 175.0	Hf 178.5	Ta 180.9	W 183.8	Re 186.2	Os 190.2	Pt 195.1	Au 197.0	Hg 200.6	Tl 204.4	Pb 207.2	Bi 209.0	Po (209)	At (210)	Rn (222)	Fr (223)	Ra 226.0	Lr (260)	Rf (261)	Db (262)	Sg (263)	Bh (262)	Hs (265)	Mt (266)	Ds (281)	Rg (280)	Cn (285)	Uut (284)	Ff (289)	Uup (288)	Lv (293)	Uus (294)	Uuo (294)																																				
Ac 227.0	Th 232.0	Pa 231.0	U 238.0	Np 237.0	Pu (244)	Am (243)	Cm (247)	Bk (247)	Cf (251)	Es (252)	Fm (257)	Md (258)	No (259)	Lr (260)	Rf (261)	Db (262)	Sg (263)	Bh (262)	Hs (265)	Mt (266)	Ds (281)	Rg (280)	Cn (285)	Uut (284)	Ff (289)	Uup (288)	Lv (293)	Uus (294)	Uuo (294)																																																					

Electronegativities of the Elements

H 2.1																					
Li 1.0	Be 1.5																				
Na 0.9	Mg 1.2															B 2.0	C 2.5	N 3.0	O 3.5	F 4.0	Ne -
K 0.8	Ca 1.0	Sc 1.3	Ti 1.5	V 1.6	Cr 1.6	Mn 1.5	Fe 1.8	Co 1.8	Ni 1.8	Cu 1.9	Zn 1.6	Ga 1.6	Ge 1.8	As 2.0	Se 2.4	Br 2.8	Kr -				
Rb 0.8	Sr 1.0	Y 1.2	Zr 1.4	Nb 1.8	Mo 1.8	Tc 1.9	Ru 2.2	Rh 2.2	Pd 2.2	Ag 1.9	Cd 1.7	In 1.7	Sn 1.8	Sb 1.9	Te 2.1	I 2.5	Xe -				
Cs 0.7	Ba 0.9	La 1.1	Hf 1.3	Ta 1.5	W 1.7	Re 1.9	Os 2.2	Ir 2.2	Pt 2.2	Au 2.4	Hg 1.9	Tl 1.8	Pb 1.8	Bi 1.9	Po 2.0	At 2.2	Rn -				
Fr 0.7	Ra 0.9	Ac 1.1																			

Activity Series of the Elements

<u>Metals</u>	Decreasing Reactivity ↘ Decreasing Reactivity ↘ Decreasing Reactivity	<u>Nonmetals</u>
Lithium		Fluorine
Potassium		Chlorine
Barium		Bromine
Strontium		Iodine
Calcium		
Sodium		
Magnesium		
Aluminum		
Manganese		
Zinc		
Chromium		
Iron		
Cadmium		
Cobalt		
Nickel		
Tin		
Lead		
Hydrogen		
Antimony		
Arsenic		
Bismuth		
Copper		
Mercury		
Silver		
Palladium		
Platinum		
Gold		

Solubility Rules

Ammonium (NH_4^+)	Salts of group IA elements are always soluble
Chlorides (Cl^-)	All soluble except AgCl , Hg_2Cl_2 , and PbCl_2 which are insoluble
Bromides (Br^-)	All soluble except AgBr , Hg_2Br_2 , HgBr_2 and PbBr_2 which are insoluble
Iodides (I^-)	All soluble except AgI , Hg_2I_2 , HgI_2 and PbI_2 which are insoluble
Fluorides (F^-)	Are generally insoluble
Chlorates (ClO_3^-), Nitrates (NO_3^-), and Acetates ($\text{C}_2\text{H}_3\text{O}_2^-$)	All soluble
Sulfates (SO_4^{2-})	Are soluble except CaSO_4 , SrSO_4 , BaSO_4 , Hg_2SO_4 , HgSO_4 , PbSO_4 , and Ag_2SO_4 which are insoluble
Phosphates (PO_4^{3-}), Carbonates (CO_3^{2-}) and Oxides (O^{2-})	Are insoluble except when with ammonium or Group IA elements
Hydroxides (OH^-)	Are insoluble except when with ammonium, Group IA elements and Group 2A elements from calcium down
Sulfides (S^{2-})	Are insoluble except when with ammonium, Group IA and 2A elements
Chromates (CrO_4^{2-})	Are generally insoluble

Name: _____

Block: _____ Date: _____

Magnet Analytical Chemistry

Lab #	Title	Did you feel comfortable with tests performed? Why? (If no come for remediation)	Remediation Date	Student Comments
Lab #	A Systems Check			
Lab #	The Punch in a Pop			
Lab #	Analysis of an Experiment			
Lab # 3	Water Analysis			
Lab # 5	Percent Water in a Hydrate			
Lab # 9	Volumetric Analysis			
Lab #	Gas Laws Lab			
Lab #	Gravimetric Lab			
Lab #	Investigation of Color & Absorbance			
Lab # 35	Spectrophotometric Metal Ion Analysis			
Lab #	A Trilogy Lab			

Lab #	A Virtual Lab: Mass Spectroscopy				
Lab #	Paper Chromatography of a Petal				
Lab #	Column Chromatography of a Spinach Leaf				
Lab # 4	Paper Chromatography				
Lab #	Electrophoresis Lab				

Name: _____ Block: _____ Date: _____

A Systems Check

Purpose:

To evaluate the functioning of the scales, and accuracy of the glassware in the lab.

Procedure #1:

1. Obtain the brass weights from your teacher.
2. Check the mass reading of both the regular balances and the analytical balances using the brass weights.
3. Record the scale checked and the reading to all available digits.
4. Determine the difference between expected and actual reading. Graph the difference and determine if a correction factor is necessary.

Data Table #1:

Balance #:	
Brass Weight	Reading

Analytical Balance #:	
Brass Weight	Reading

How accurate is the balance? Does the balance need a \pm recording? Is the change consistent over the entire set of masses?

Procedure #2:

1. Obtain beakers 4 beakers of different sizes, 3 graduated cylinders of different sizes and set up a buret on a ring stand.
2. Obtain approximately 100 ml of distilled water. (Re-use the water for all testing.)
3. Place a piece of glass ware on one of the balances that you evaluated in procedure #1. Tare the balance.
**for the buret place a beaker on the balance and under the buret.
4. Pour water in until the glass ware is one quarter full, half full and two thirds full (Or 80.00 grams maximum) record the reading on the balance and the glassware, remember to record all possible digits.
5. Repeat process for all glassware.
6. Evaluate the glassware for accuracy.

Glassware: \pm	
Mass	Volume

Glassware: \pm	
Mass	Volume

Glassware: \pm	
Mass	volume

Glassware:	
±	
Mass	Volume
Glassware:	
±	
Mass	Volume

Glassware:	
±	
Mass	Volume
Glassware:	
±	
Mass	Volume

Glassware:	
±	
Mass	volume
Glassware:	
±	
Mass	volume

Would you agree with the accuracy rating that was recorded on the glassware?

Procedure #3:

1. Place approximately 25 ml of distilled water into the buret.
2. Place a small beaker on the balance under the buret. Tare the balance.
3. Slowly open the buret stop cock allowing only 5 drops of water to be released.
4. Record the mass and the change in volume.
5. Repeat four times.
6. Average the mass and the volume for 5 drops of water from the buret.

5 drops of water from buret.	
Mass	Volume
Average mass:	Average volume:

Name: _____ Block: ____ Date: _____

The “Punch” in a Pop

Purpose: The purpose of this lab is to calculate the percent water in the composition of a popcorn kernel and the amount of pressure required to cause a popcorn kernel to pop. Analysis of possible errors will also be performed along with recommendations for improvements will be considered. Calculations will be made indirectly by measuring the changes in a bag of microwave popcorn. (Products of the lab maybe eaten in class.)

Data Table: (be sure to record all appropriate significant figures!)

Mass of Unpopped Corn and Bag	g
Mass of Popped Corn and Bag	g
Mass of Empty Bag	g
Mass of Unpopped Corn	g
Mass of Water Lost	g
Length of bag	cm
Height of Bag	cm
Width of Bag	cm
Volume of Bag	cm ³
Percent Water in Popcorn	%
Moles of Water Lost	mol

Calculations:

1. Calculate the mass of unpopped corn for your popcorn and record in data table.
 2. Calculate the mass of water lost for your popcorn and record in data table.
 3. Calculate the percent water in your popcorn sample.
 4. If the actual percentage of water in popcorn is 13.7%, what is your percent error?
 5. Calculate the pressure (in atm) of the water vapor at the time of “pop,” use the equation:
 $PV = nRT$ Remember $R=0.0821 \frac{\text{L atm}}{\text{mol K}}$
- Hint: Assume that the popcorn pops at the boiling point of cooking oil (225°C).
6. If the actual pressure of the pop is 9.1 atm, what is your percent error?

Discussions:

1. Why might your percent water have been different than the expected value? If the percent water is too high how could this effect the popcorn? If the percent water is too low how could this effect the popcorn? As you discuss these keep in mind how the two parts of the lab are related (% water and pressure).
2. What could you have done to improve the data collected in this lab? (Please give at least two specific improvements and detailed responses.)
3. What other aspects about the popcorn could have affected your results. (Please give at least two specific aspects and be detailed with your responses.)

Evaluation of someone else's experiment.

Today we will watch a small snippet of a MythBusters episode from Episode 23, November 16, 2004. The episode is about talking and plant growth. As you watch the experiment record what you feel is the hypothesis, testing method, control and constants. What do you feel should have been treated as a constant that was not? Was the control used appropriate, why or why not? Was the testing method appropriate, why or why not? Did the experiment ultimately support the hypothesis? What, if any, changes would you have made? What experiment could be done to support the one done by Mythbusters? What experiments could be done as an off-shoot?

Seven small greenhouses were set up on the M5 Industries roof. Four were set up with stereos playing endlessly looping recordings (as having the Mythbusters actually talk to the plants could contaminate the samples with their expelled carbon dioxide): Two of negative speech, two of positive speech (Kari and Scottie each made one positive and one negative soundtrack), a fifth with classical music and a sixth with intense death metal music. A seventh greenhouse, used as a control sample, had no stereo. The greenhouses with the recordings of speech grew better than the control, regardless of whether such talk was kind or angry. The plants in the greenhouse with the recording of classical music grew better, while the plants in the greenhouse with the recording of intense death metal grew best of all.



Experiment 3

Water Analysis: Solids

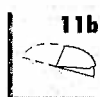
"Clear" water from streams contains small quantities of dissolved and suspended solids.

- To determine the total, dissolved, and suspended solids in a water sample
- To determine the ions present in the solids of a water sample.

OBJECTIVES

The following techniques are used in the Experimental Procedure:

TECHNIQUES



Surface water is used as the primary drinking water source for many large municipalities. The water is piped into a water treatment facility where impurities are removed and bacteria are killed before the water is placed into the distribution lines. The contents of the surface water must be known and predictable so that the treatment facility can properly and adequately remove these impurities. Tests are used to determine the contents of the surface water.

Water in the environment has a large number of impurities with an extensive range of concentrations. **Dissolved solids** are water-soluble substances, most often salts although some dissolved solids may come from organic sources. Naturally occurring dissolved salts generally result from the movement of water over or through mineral deposits, such as limestone. These dissolved solids, characteristic of the **watershed**, generally consist of the sodium, calcium, magnesium, and potassium cations and the chloride, sulfate, bicarbonate, carbonate, bromide, and fluoride anions. Anthropogenic (human-related) dissolved solids include nitrates from fertilizer runoff and human wastes, phosphates from detergents and fertilizers, and organic compounds from pesticides, sewage runoff, and industrial wastes.

Dissolved salts can be problematic in potable water. Typical dissolved salt concentrations range from 20 to 1000 mg/L, although most waters are less than 500 mg/L. High concentrations also indicate "hard" water (Experiment 21) which can clog pipes and industrial cooling systems. Also, high concentrations of dissolved salts can cause diarrhea or constipation in some people.

Salinity, a measure of the total salt content in a water sample, is expressed as the grams of dissolved salts per kilogram of water or as **parts per thousand** (ppt). The average ocean salinity is 35 ppt whereas fresh water salinity is usually less than 0.5 ppt. Brackish water, where fresh river water meets salty ocean water, varies from 0.5 ppt to 17 ppt. For water samples with low organic levels, the salinity of a water sample approximates that of total dissolved solids (TDS) content.

More specifically, the anions that account for the salinity of the water are generally the carbonates and bicarbonates, CO_3^{2-} and HCO_3^- , the halides (Cl^- , Br^- , and I^-), the

INTRODUCTION

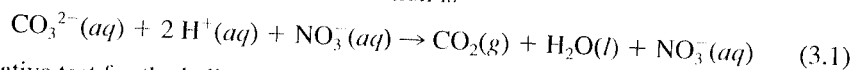
Watershed: the land area from which the natural drainage of water occurs

Parts per thousand (ppt): 1g of substance per 1000g (1 kg) of sample

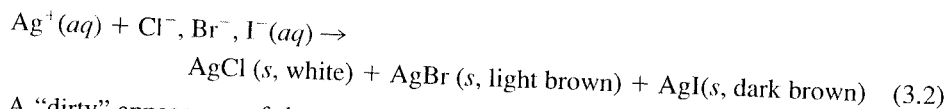
Ionic equation: ionic equations, though appearing somewhat premature in this manual, are written to better illustrate the ions in solution that are involved in the chemical reactions and observations. Experiment 6 will further illustrate the use of ionic equations for chemical reactions.

phosphates, PO_4^{3-} , and the sulfates, SO_4^{2-} . A qualitative testing of a water sample can determine the presence of these various ions.

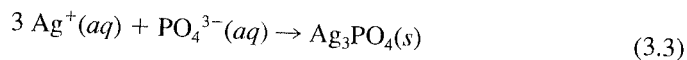
To test for the presence of carbonates and bicarbonates in the water, an acid, generally nitric acid, HNO_3 , is added to the sample resulting in the evolution of carbon dioxide gas. The **ionic equation** for the reaction is



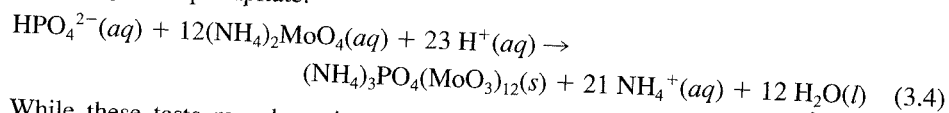
A qualitative test for the halides is the addition of silver ion, resulting in a silver halide precipitate:



A "dirty" appearance of the precipitate is evidence for a mixture of the halides in the water. Additionally, if the phosphates are present, a white silver phosphate precipitate forms:

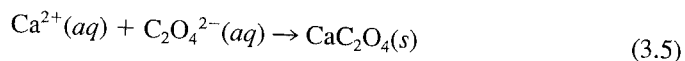


The presence of phosphate ion (or HPO_4^{2-}) can also be determined independently by the addition of an ammonium molybdate solution to the sample, resulting in the formation of a yellow precipitate:



While these tests may have interferences as performed in this experiment, a more detailed experimental and systematic procedure is presented in Experiment 37.

The most common cation present in "natural" waters is calcium. Calcium is the principal ion responsible for water hardness. See Experiment 21. A qualitative test for its presence is its formation of insoluble calcium oxalate.



Suspended solids are very finely divided particles, kept in suspension by the turbulent action of the moving water, are *insoluble* in water but are filterable. Total suspended solids (TSS) is a measure of the turbidity and the clarity of the water.

High concentrations of suspended solids, such as decayed organic matter, sand, silt, and clay, can settle to cover (and suffocate) the existing ecosystem at the bottom of a lake, can make disinfectants for water treatment less effective, and can adsorb/adsorb various organic and inorganic pollutants resulting in an increase of their residence in the water sample.

Total solids (TS) are the sum of the dissolved and suspended solids in the water sample. In this experiment the total solids and the dissolved solids are determined directly; the suspended solids are assumed to be the difference, since

$$\text{total solids (TS)} = \text{total dissolved solids (TDS)} + \text{total suspended solids (TSS)} \quad (3.6)$$

The U.S. Public Health Service recommends that drinking water not exceed 500 mg total solids/kg water, or 500 **ppm**. However, in some localities, the total solids content may range up to 1000 mg/kg of potable water; that's 1 g/L!! An amount over 500 mg/kg water does not mean the water is unfit for drinking; an excess of 500 mg/kg is merely not recommended.

Suspended solids: solids that exhibit colloidal properties or solids that remain in the water because of turbulence

ppm: 1 mg substance per kilogram sample = 1 part per million (ppm)

EXPERIMENTAL PROCEDURE

Filtrate: the solution that passes through the filter into a receiving flask.

Procedure Overview: The amounts of total, dissolved, and suspended solids in a water sample are determined in this experiment. The water sample is filtered to remove the suspended solids and the **filtrate** is evaporated to dryness to determine the dissolved solids. The water sample may be from the ocean, a lake, a stream, or from an underground aquifer.



Figure 3.1 Wash the splattered material from the convex side of the watch glass.

Obtain 100 mL of a water sample from your instructor. Preferably the water sample is high in **turbidity**. Record the sample number and write a short description of the sample on the Report Sheet. With approval, bring your own “environmental” water sample to the laboratory for analysis. Ask your instructor whether evaporating dishes or 250 mL beakers are to be used for the analysis.

If time allows, the experiment should be repeated twice. A basis for water quality is *not* determined from a single analysis—a minimum of three trials is necessary for reputable analytical data. Ask your instructor for additional information.

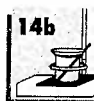
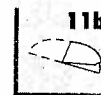
Assume the density of your water sample to be 1.02 g/mL.

Turbid sample: a cloudy suspension due to stirred sediment.



1. **Filter the Water Sample.** Gravity filter about 50 mL of a *thoroughly stirred or shaken* water sample into a clean, dry 100-mL beaker. While waiting for the filtration to be completed, proceed to Part B.
2. **Evaporate the Filtrate to Dryness.**
 - a. Clean, dry, and measure the mass (± 0.001 g) of an evaporating dish (or 250-mL beaker).
 - b. Pipet a 25-mL aliquot (portion) of the filtrate into the evaporating dish (250-mL beaker). Determine the combined mass of the sample and evaporating dish (beaker).
 - c. Use a hot plate or direct flame (Figure T.14a or T.14b) to *slowly* heat—do not boil—the mixture to dryness.
 - d. As the mixture nears dryness, cover the evaporating dish (beaker) with a watch glass and reduce the intensity of the heat.¹ If spattering occurs, allow the dish to cool to room temperature, rinse the adhered solids from the watch glass (see Figure 3.1) and return the rinse to the dish.
3. **A Final Heating to Dryness.** Again heat slowly, being careful to avoid further spattering. After all of the water has evaporated, reduce the heat of the hot plate, or maintain a “cool” flame beneath the dish for 3 minutes. Allow the dish to cool to room temperature and determine its final mass. Cool the evaporating dish and sample in a desiccator, if available.

A. Total Dissolved Solids (TDS)



Cool flame: a Bunsen flame of low intensity—a slow rate of natural gas is flowing through the burner barrel.

¹This reduces the spattering of the remaining solid and its subsequent loss in analysis.

B. Total Solids (TS) and Total Suspended Solids (TSS)



1. **Evaporate an Original Water Sample to Dryness.** a. Clean, dry, and measure the mass (± 0.001 g) of a second evaporating dish (or 250-mL beaker).
b. Thoroughly stir or agitate 100 mL of the original water sample; pipet² a 25-mL aliquot of this sample into the evaporating dish (250-mL beaker). Record the combined mass of the water sample and evaporating dish (beaker).
c. Evaporate *slowly* the sample to dryness as described in Part A.2. Record the mass of the solids remaining in the evaporating dish.
2. **Total Suspended Solids.** Collect the appropriate data to determine the total suspended solids in the water sample.

C. Analysis of Data

Appendix B



1. **Precision of Data?** Compare your TDS and TSS data with three other chemists in your laboratory who have analyzed the *same* water sample. Record their results on the Report Sheet. Calculate the average value for the TSS in the water sample.

D. Chemical Tests³



1. **Test for Carbonates and Bicarbonates.** With your spatula loosen small amounts of the dried samples from Part A and Part B and transfer each to *separately* marked 75-mm test tubes. Add several drops of 6 M HNO₃ (**Caution:** HNO₃ is corrosive and a severe skin irritant) and observe. What can you conclude from your observation?
2. **Test for Chlorides (Halides).** To each of the test tubes add 10 drops of water, agitate the solution, and add several drops of 0.01 M AgNO₃ (**Caution:** AgNO₃ is a skin irritant) and observe. What can you conclude from your observation?
3. **Test for Phosphates.** With your spatula loosen small amounts of the dried sample from Parts A and B and transfer each to *separately* marked 75-mm test tubes. To each test tube add ~10 drops of water and several drops of 6 M HNO₃ (**Caution:** Avoid skin contact. Do not inhale fumes.) followed by 1 mL of 0.5M (NH₄)₂MoO₄. Shake and warm the resulting solutions in a warm water bath (~60°C, Figure T.13b) and let stand for 10–15 minutes. The yellow color can be slow to form if PO₄³⁻ is present in the sample.
4. **Test for Calcium Ion.** With your spatula loosen small amounts of the dried samples from Parts A and B and transfer each to *separately* marked 75-mm test tubes. Add about 10 drops of water, agitate the solution, and add several drops of 1 M K₂C₂O₄ and observe. What can you conclude from your observation?

Appendix G



Disposal: Discard the dried salts from Part A and B and the test solutions from Part D in the "Waste Salts" container.

CLEANUP: Rinse the test tubes and evaporating dishes (250-mL beakers) with tap water and twice with deionized water.

The Next Step

Devise a plan to determine the changes in TSS and TDS at various points along a water source (river, stream, lake, drinking water, etc.). Explain why the values change as a result of location, rainfall, season, time of day, etc. Test to determine the ions that are primary contributors to the TDS of the sample.

²If the solution appears to be so turbid that it may plug the pipet tip, use a 25-mL graduated cylinder to measure the water sample as accurately as possible.

³For each of the tests in Part D there are other ions that may show a positive test. However the ions being tested are those most common in environmental water samples.

Experiment 3 *Prelaboratory Assignment*

Water Analysis: Solids

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

1. List several anions, by formula, that contribute to the salinity of a water sample.

2. Distinguish between and characterize the “total dissolved solids” and “total suspended solids” in a water sample.

3. Experimental Procedure, Part A.2c. Explain why a “cool flame” is important to use in heating a solution to dryness.

4. a. What is an **aliquot** of a sample?

b. What is the **filtrate** in a gravity filtration procedure?

c. How full (the maximum level) should a funnel be filled with solution in a filtration procedure?



5. Experimental Procedure, Part D. What observation is “expected” when
 - a. an acid (nitric acid, HNO_3) is added to a solution containing carbonate or bicarbonate ions? See Experiment 2, Experimental Procedure, Part A.3.

b. silver ion is added to a solution containing chloride (or bromide or iodide) ions? See Appendix G.

c. a solution of 0.5 M $(\text{NH}_4)_2\text{MoO}_4$ is added to a water sample containing phosphate ion (and heated)? Explain.

6. The following data were collected for determining the concentration of suspended solids in a water sample (density = 1.02 g/mL).

	<i>Trial 1</i>	<i>Trial 2</i>	<i>Trial 3</i>	<i>Trial 4</i>	<i>Trial 5</i>	<i>Trial 6</i>
Volume of sample (mL)	25.0	20.0	50.0	25.0	20.0	25.0
Mass of sample (g)						
Mass of dry solid (g)	10.767	8.436	21.770	10.826	8.671	10.942
Mass of solid/mass of sample (g/g)						

a. What is the *average* total suspended solids (TSS) in the water sample? Express this measurement in ppt (parts per thousand, g/kg). See Appendix B.

*b. Calculate the standard deviation and the relative standard deviation (% RSD) for the analyses.

7. A 25.0-mL aliquot of a well-shaken sample of river water is pipetted into a 25.414-g evaporating dish. After the mixture is evaporated to dryness, the dish and dried sample has a mass of 36.147 g. Determine the total solids in the sample; express total solids in units of g/kg sample (parts per thousand, ppt). Assume the density of the river water to be 1.01 g/mL.

Water Analysis: Solids

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

Sample Number: _____ Describe the nature of your water sample, i.e., its color, turbidity, etc.

A. Total Dissolved Solids (TDS)

Trial 1

1. Mass of evaporating dish (beaker) (g) _____
2. Mass of water sample plus evaporating dish (beaker) (g) _____
3. Mass of water sample (g) _____
4. Mass of *dried* sample (g) _____
5. Mass of dissolved solids in 25-mL aliquot of filtered sample (g) _____
6. Mass of dissolved solids per total mass of sample (g solids/g sample) _____
7. Total dissolved solids (TDS) or salinity (g solids/kg sample, ppt) _____

B. Total Solids (TS) and Total Suspended Solids (TSS)

1. Mass of evaporating dish (beaker) (g) _____
2. Mass of water sample plus evaporating dish (beaker) (g) _____
3. Mass of water sample (g) _____
4. Mass of *dried* sample (g) _____
5. Mass of total solids in 25-mL aliquot of unfiltered sample (g) _____
6. Mass of total solids per total mass of sample (g solids/g sample) _____
7. Total solids (g solids/kg sample, ppt) _____
8. Total suspended solids (g solids/kg sample, ppt) _____

C. Analysis of Data

Chemist No.	#1 (you)	#2	#3	#4
TDS (g/kg)				
TS (g/kg)				
TSS (g/kg)				

Average value of total suspended solids (TSS) from four chemists (\bar{x}) = _____

D. Chemical Tests.

Test	Observation	Conclusion
1. CO_3^{2-} , HCO_3^- (TDS)	_____	_____
CO_3^{2-} , HCO_3^- (TS)	_____	_____
2. Cl^- , Br^- , I^- (TDS)	_____	_____
Cl^- , Br^- , I^- (TS)	_____	_____
3. PO_4^{3-} (TDS)	_____	_____
PO_4^{3-} (TS)	_____	_____
4. Ca^{2+} (TDS)	_____	_____
Ca^{2+} (TS)	_____	_____

Write a summary of your assessment of the quality of your water sample.

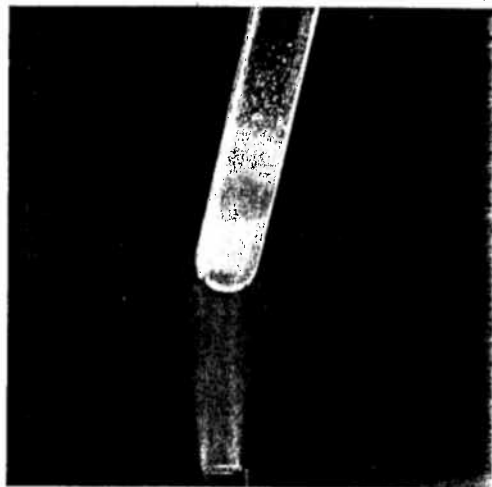
Laboratory Questions

Circle the questions that have been assigned.

1. Part A.2. The evaporating dish was not properly cleaned of a volatile material before its mass was determined. When the sample is heated to dryness the volatile material is removed. How does this error in technique affect the reported TDS for the water sample? Explain.
2. Part A.2. Some spattering of the sample onto the watchglass does occur near dryness. In a hurry to complete the analysis, the chemist chooses not to return the spattered solids to the original sample and skips the first part of Part A.3. Will the reported TDS for the water sample be too high or too low? Explain.
3. Part A.3. The sample in the evaporating dish is *not* heated to total dryness. How will this error in technique affect the reported value for TDS? Explain. TSS? Explain.
4. Part A.3. As the sample cools moisture from the atmosphere condenses on the outside of the evaporating dish (beaker) before the mass is measured. How does the presence of the condensed moisture affect the reported TDS for the water sample? Explain.
5. Part B.1. The sample in the evaporating dish (beaker) is *not* heated to total dryness. How will this error in technique affect the reported value for total solids? Explain. TSS? Explain.
6. Parts A and B. Suppose the water sample has a relatively high percent of volatile solid material. How would this have affected the reported mass of
 - a. dissolved solids? Explain.
 - b. total solids? Explain.
 - c. suspended solids? Explain.
7. Part D.2. When several drops of 0.010 M AgNO_3 are added to a test sample, a white precipitate forms. What can you conclude from this observation? Explain.
8. Part D.3. a. When 1 mL of $0.5\text{ M (NH}_4)_2\text{MoO}_4$ is added to the water sample, a yellow solution is observed but no precipitate. What can you conclude from this observation? Explain.
 - b. How does this observation help in interpreting any observations from Part D.2?

Experiment 5

Percent Water in a Hydrated Salt



Heat readily removes the hydrated water molecules (top of sample in test tube) from copper(II) sulfate pentahydrate forming anhydrous copper(II) sulfate (bottom).

- To determine the percent by mass of water in a hydrated salt
- To learn to handle laboratory apparatus without touching it

OBJECTIVES

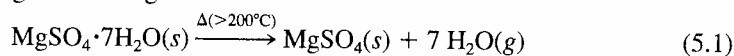
The following techniques are used in the Experimental Procedure

TECHNIQUES



Many salts occurring in nature, purchased from the grocery shelf or from chemical suppliers are **hydrated**; that is, a number of water molecules are chemically bound to the ions of the salt in its crystalline structure. These water molecules are referred to as **waters of crystallization**. The number of moles of water per mole of salt is usually a constant. For example, iron(III) chloride is purchased as $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, not as FeCl_3 , and copper(II) sulfate as $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, not as CuSO_4 . For some salts, the water molecules are so weakly bound to the ions that heat removes them to form the **anhydrous** salt. Hydrated salts that spontaneously (without heat) lose water molecules to the atmosphere are **efflorescent**, whereas salts that readily absorb water are **deliquescent**.

For Epsom salt (magnesium sulfate heptahydrate, Figure 5.1), the anhydrous salt, MgSO_4 , forms with gentle heating.



In other salts such as $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, the water molecules are so strongly bound to the salt that anhydrous FeCl_3 cannot form regardless of the intensity of the heat.

In Epsom salt 7 moles of water, or 126.1 g of H_2O , are bound to each mole of magnesium sulfate, or 120.4 g of MgSO_4 . The percent by mass of water in the salt is

$$\frac{126.1 \text{ g H}_2\text{O}}{(126.1 \text{ g} + 120.4 \text{ g}) \text{ salt}} \times 100 = 51.16\% \text{ H}_2\text{O} \quad (5.2)$$

A **gravimetric analysis** is an analytical method that relies almost exclusively on mass measurements for the analysis. Generally the substance being analyzed must have a mass large enough to be measured easily with the balances that are available in the laboratory.

This experiment uses the gravimetric analysis method to determine the percent by mass of water in a hydrated salt. The mass of a hydrated salt is measured, the sample is heated to drive off the hydrated water molecules (the waters of crystallization), and then the mass of remaining sample is measured again. Cycles of heating and measuring of the sample's mass are continued until reproducibility of the mass measurements is attained.

INTRODUCTION

Hydrate: water molecules are chemically bound to the ions of the salt as part of the structure of the compound

Anhydrous: without water



Figure 5.1 Epsom salt, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

EXPERIMENTAL PROCEDURE



Procedure Overview: The mass of a hydrated salt is measured before and after it is heated to a high temperature. This mass difference and the mass of the anhydrous salt are the data needed to calculate the percent water in the original hydrated salt.

You are to complete *at least* two trials in this experiment. Obtain a hydrated salt from your instructor. Record the unknown number of your hydrated salt on the Report Sheet. A 150-mm test tube may be substituted for the crucible and lid. Ask your instructor.

A. Sample Preparation



Fired: heated to a very high temperature to volatilize impurities



Figure 5.2

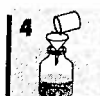
Handle the crucible with tongs after heating.

1. **Prepare a Clean Crucible.** Obtain a clean crucible and lid. Check the crucible for stress fractures or fissures, which are common in often-used crucibles. If none are found, support the crucible and lid on a clay triangle and heat with an intense flame for 5 minutes (see Technique 15C, Figure T.15c). Allow them to cool slowly. (**Caution:** Do not set them on the lab bench for fear of contamination.)^{1,2}

Caution: hot and cool crucibles look the same—do not touch! Determine the mass (± 0.001 g) of the **fired**, cool crucible and lid and record. Handle the crucible and lid with the crucible tongs for the remainder of the experiment (Figure 5.2); do *not* use your fingers—oil from the fingers can contaminate the surface of the crucible and lid. Be sure the crucible tongs are clean and dry.

2. **Determine the Mass of Sample.** Add no more than 3 g of your hydrated salt to the crucible. Measure and record the combined mass (± 0.001 g) of the crucible, lid, and hydrated salt. Calculate the mass of the hydrated salt.
3. **Adjust the Crucible Lid.** Return the crucible (use crucible tongs only) with the sample to the clay triangle; set the lid just off the lip of the crucible to allow the evolved water molecules to escape on heating (Figure T.15e).

B. Thermal Decomposition of the Sample



1. **Heat the Sample.** Initially heat the sample slowly and then gradually intensify the heat. Do *not* allow the crucible to become red hot. This may cause the anhydrous salt to decompose as well. Maintain the high temperature on the sample for 10 minutes.

Cover the crucible with the lid; allow them to cool to room temperature (see footnote 1). Determine the combined mass of the crucible, lid, and anhydrous salt on the same balance that was used for earlier measurements.

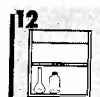
2. **Have You Removed All of the Water?** Reheat the sample for 2 minutes (Figure T.15e), but do *not* intensify the flame—avoid the decomposition of the salt. Cool to room temperature and again measure the combined mass. If this second mass measurement of the anhydrous salt disagrees by greater than ± 0.010 g from that in Part B.1, repeat Part B.2.
3. **Repeat with a New Sample.** Repeat the experiment two more times with original hydrated salt samples.

Disposal: Dispose of all waste anhydrous salt in the "Waste Solids" container.

CLEANUP: Rinse the crucible with 2–3 milliliters of 1 M HCl and discard in the "Waste Acids" container. Then rinse several times with tap water and finally deionized water. Each water rinse can be discarded in the sink.

The Next Step

(1) Most any substance has water as a part of its composition, either adsorbed or bonded to the substance. Develop a procedure for a determining the percent water in a soil sample . . . remember that dissolved gases may also be present. (2) How rapidly do potato chips or crackers absorb water from the atmosphere? Develop a data plot of percent water vs. time for "soggy crackers." (3) A coal analysis involves the determination of the (a) percent moisture, (b) percent volatile combustible matter, VCM, (c) percent ash, and (d) percent fixed carbon, FC, where $\%FC = 100\% - (\%H_2O + \%VCM + \%ash)$. Develop a procedure for the analysis for a coal sample . . . the analysis indicates the quality of the coal for combustion, coking, etc.



¹Place the crucible and lid in a desiccator (if available) for cooling.

²If the crucible remains dirty after heating, move the apparatus to the fume hood, add 1–2 mL of 6 M HNO_3 , and gently evaporate to dryness. (**Caution:** Avoid skin contact, flush immediately with water.)

Experiment 5 *Prelaboratory Assignment*

Percent Water in a Hydrated Salt

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

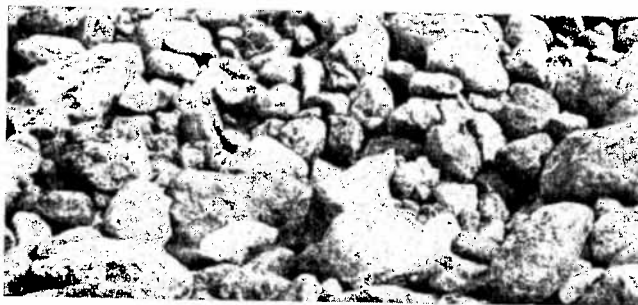
1. Calcium chloride, a deliquescent salt, is used as a desiccant in laboratory desiccators. Explain.



2. Experimental Procedure, Part A.1. What is the purpose of firing the crucible?

3. A 1.994-g sample of gypsum, a hydrated salt of calcium sulfate, CaSO_4 , is heated at a temperature greater than 170°C in a crucible until a constant mass is reached. The mass of the anhydrous CaSO_4 salt is 1.577 g.

Calculate the percent by mass of water in the hydrated calcium sulfate salt.



4. The gravimetric analysis of this experiment is meant to be quantitative; therefore, all precautions should be made to minimize errors in the analysis.
- a. The crucible and lid are handled exclusively with crucible tongs in the experiment. How does this technique maintain the integrity of the analysis?



- b. Mass measurements of the crucible, lid, and sample are performed only at room temperature. Why is this technique necessary for a gravimetric analysis?

- c. Why is the position of the crucible lid critical to the dehydration of the salt during the heating process? Explain.

5. The following data were collected from the gravimetric analysis of a hydrated salt:

Mass of crucible and lid (g)	19.437
Mass of crucible, lid, and hydrated salt (g)	21.626
Mass of crucible, lid, and anhydrous salt (g)	21.441

Determine the percent water in the hydrated salt.

6. a. What is the percent by mass of water in copper(II) sulfate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$? See opening photo.

b. What mass due to waters of crystallization is present in a 3.38-g sample of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$?

Experiment 5 Report Sheet

Percent Water in a Hydrated Salt

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

Unknown No. _____

	<i>Trial 1</i>	<i>Trial 2</i>	<i>Trial 3</i>
1. Mass of crucible and lid (g)	_____	_____	_____
2. Mass of crucible, lid, and hydrated salt (g)	_____	_____	_____
3. Mass of crucible, lid, and anhydrous salt	_____	_____	_____
1st mass measurement (g)	_____	_____	_____
2nd mass measurement (g)	_____	_____	_____
3rd mass measurement (g)	_____	_____	_____
4. Final mass of crucible, lid, and anhydrous salt (g)	_____	_____	_____

Calculations

1. Mass of hydrated salt (g)	_____	_____	_____
2. Mass of anhydrous salt (g)	_____	_____	_____
3. Mass of water lost (g)	_____	_____	_____
4. Percent by mass of volatile water in hydrated salt (%)	_____*	_____*	_____*
5. Average percent H ₂ O in hydrated salt (%H ₂ O)	_____*		
6. Standard deviation of %H ₂ O	_____*		
7. Relative standard deviation of %H ₂ O in hydrated salt (%RSD)	_____*		

*Show calculations on next page.

*Calculations for Trial 1. Show your work.

*Calculation of Standard Deviation and %RSD. Appendix B.

Laboratory Questions

Circle the questions that have been assigned.

1. Part A.1. During the cooling of the fired crucible, water vapor condensed on the crucible wall before its mass measurement. The condensation did not occur following thermal decomposition of the hydrated salt in Part B. Will the reported percent water in the hydrated salt be reported too high or too low? Explain.
2. Part A.1. The fired crucible is handled with (oily) fingers before its mass measurement. Subsequently in Part B.1, the oil from the fingers is burned off. How does this technique error affect the reported percent water in the hydrated salt? Explain.
3. Part A.1. The crucible is handled with (oily) fingers after its mass measurement but before the ~ 3 g sample of the hydrated salt is measured (Part A.2). Subsequently in Part B.1, the oil from the fingers is burned off. How does this technique error affect the reported percent water in the hydrated salt? Explain.
4. Part A.1. Suppose the original sample is unknowingly contaminated with a second anhydrous salt. Will the reported percent water in the hydrated salt be too high, too low, or unaffected by its presence? Explain.
- *5. After heating the crucible in Part A.1, the crucible is set on the lab bench where it is contaminated with the cleaning oil used to clean the lab bench but before its mass is measured. The analysis continues through Part B.1 where the mass of the anhydrous salt is determined. While heating, the cleaning oil is burned off the bottom of the crucible. Describe the error that has occurred; that is, is the mass of the anhydrous salt remaining in the crucible reported as being too high or too low? Explain.
6. Part B.1. The hydrated salt is overheated and the anhydrous salt thermally decomposes, one product being a gas. Will the reported percent water in the hydrated salt be reported too high, too low, or be unaffected? Explain.
7. Part B.2. Because of a lack of time, Bill decided to skip this step in the Experimental Procedure. Will his haste in reporting the "percent H_2O in the hydrated salt" likely be too high, too low, or unaffected? Explain.

Name: _____

Calcium Analysis by EDTA Titration

PRESTUDY

1. A 0.4505 g sample of CaCO_3 was dissolved in HCl and the resulting solution was diluted to 250.0 mL in a volumetric flask. A 25.00 mL aliquot of the solution required 24.25 mL of an EDTA solution for titration to the Eriochrome Black T end point.

a. How many moles of CaCO_3 were used?

b. What is the concentration (molarity) of Ca^{2+} in the 250.0 mL of CaCl_2 solution?

c. How many moles of Ca^{2+} are contained in a 25.00 mL sample?

d. How many moles of EDTA are contained in the 24.25 mL used for the titration?

e. What is the concentration (molarity) of the EDTA solution?

2. If 100.00 mL of a water sample required 23.24 mL of EDTA of the concentration found in part e of problem 1, what is the hardness of the water in terms of ppm CaCO_3 (ppm = mg/L)?

Experiment 8

Limiting Reactant

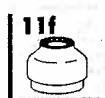


Calcium oxalate crystals contribute to the formation of kidney stones.

- To determine the limiting reactant in a mixture of two soluble salts
- To determine the **percent composition** of each substance in a salt mixture

OBJECTIVES

The following techniques are used in the Experimental Procedure

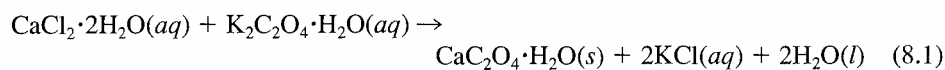


TECHNIQUES

Percent composition: the mass ratio of a component of a mixture or compound to the total mass of the sample times 100

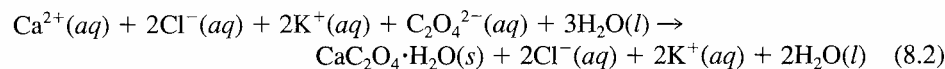
Two factors affect the yield of products in a chemical reaction: (1) the amounts of starting materials (reactants) and (2) the **percent yield** of the reaction. Many experimental conditions, for example, temperature and pressure, can be adjusted to increase the yield of a desired product in a chemical reaction, but because chemicals react according to fixed mole ratios (**stoichiometrically**), only a limited amount of product can form from measured amounts of starting materials. The reactant determining the amount of product generated in a chemical reaction is called the **limiting reactant** in the chemical system.

To better understand the concept of the limiting reactant, let us look at the reaction under investigation in this experiment, the reaction of calcium chloride dihydrate, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, and potassium oxalate monohydrate, $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, in an aqueous solution.



Calcium oxalate monohydrate, $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$, is an insoluble compound, but is found naturally in a number of diverse locations. It is found in plants, such as rhubarb leaves, agave, and (in small amounts) spinach, and is the cause of most kidney stones. In small doses, it causes a severe reaction to the lining of the digestive tract. However, the handling of calcium oxalate in the laboratory is safe, so long as it isn't transferred to the mouth.

For the reaction system in this experiment, both the calcium chloride and potassium oxalate are soluble salts but the calcium oxalate is insoluble. The **ionic equation** for the reaction is



INTRODUCTION

Percent yield:

$$\left(\frac{\text{actual yield}}{\text{theoretical yield}} \right) \times 100$$

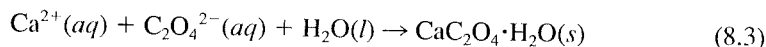
Stoichiometrically: by a study of a chemical reaction using a balanced equation

Ionic equation: A chemical equation that presents ionic compounds in the form in which they exist in aqueous solution. See Experiment 6.

Spectator ions: cations or anions that do not participate in any observable or detectable chemical reaction

Net ionic equation: an equation that includes only those ions that participate in the observed chemical reaction

Presenting only the ions that show evidence of a chemical reaction (i.e., the formation of a precipitate) and by removing the **spectator ions** (i.e., no change of ionic form during the reaction), we have the **net ionic equation** for the observed reaction.



Calcium oxalate monohydrate is thermally stable below $\sim 90^\circ\text{C}$, but forms the anhydrous salt at temperatures above 110°C .

Therefore, one mole of Ca^{2+} (from one mole of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, molar mass = 147.02 g/mol) reacts with one mole of $\text{C}_2\text{O}_4^{2-}$ (from one mole of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, molar mass = 184.24 g/mol) to produce one mole of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (molar mass = 146.12 g/mol). If the calcium oxalate is heated to temperatures greater than 110°C for drying, then anhydrous CaC_2O_4 (molar mass = 128.10 g/mol) is the product.

In Part A of this experiment the solid salts $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ form a heterogeneous mixture of unknown composition. The mass of the solid mixture is measured and then added to water—insoluble $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ forms. The $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate is collected, via gravity filtration and dried, and its mass is measured.

The percent composition of the salt mixture is determined by first testing for the limiting reactant. In Part B, the limiting reactant for the formation of solid calcium oxalate monohydrate is determined from two precipitation tests of the solution: (1) the solution is tested for an excess of calcium ion with an oxalate reagent—observed formation of a precipitate indicates the presence of an excess of calcium ion (and a limited amount of oxalate ion) in the salt mixture; (2) the solution is also tested for an excess of oxalate ion with a calcium reagent—observed formation of a precipitate indicates the presence of an excess of oxalate ion (and a limited amount of calcium ion) in the salt mixture.

Calculations



The calculations for the analysis of the salt mixture require some attention. “How do I proceed to determine the percent composition of a salt mixture of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ by only measuring the mass of the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate?”

Example: A 0.538-g sample of the salt mixture is added to water and after drying (to less than 90°C) 0.194 g of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ is measured. Tests reveal that $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ is the limiting reactant. What is the percent composition of the salt mixture? How many grams of the excess $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ were in the salt mixture?

Solution: Since $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ is the limiting reactant, then, according to Equation (8.1), the moles of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in the salt mixture equals the moles of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ formed. Therefore, the calculated mass of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in the original salt mixture is

$$\begin{aligned} \text{grams } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O} &= 0.194 \text{ g } \text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O} \times \frac{\text{mol } \text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}}{146.12 \text{ g } \text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} \\ &\times \frac{1 \text{ mol } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}}{1 \text{ mol } \text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} \times \frac{184.24 \text{ g } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}}{1 \text{ mol } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}} \\ &= 0.245 \text{ g } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O} \text{ in the salt mixture.} \end{aligned}$$

The percent by mass of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in the original salt mixture is

$$\% \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O} = \frac{0.245 \text{ g } \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}}{0.538 \text{ g sample}} \times 100 = 45.5\% \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$$

The mass of the $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in the salt mixture is the difference between the mass of the sample and the mass of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ or ($0.538 \text{ g} - 0.245 \text{ g} =$) 0.293 g. The percent by mass of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in the original salt mixture is

$$\% \text{CaCl}_2 \cdot 2\text{H}_2\text{O} = \frac{0.538 \text{ g} - 0.245 \text{ g}}{0.538 \text{ g sample}} \times 100 = 54.5\% \text{CaCl}_2 \cdot 2\text{H}_2\text{O}$$

According to Equation 8.1, the moles of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ that react equals the moles of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (the limiting reactant) that react equals the moles of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ that precipitate. Therefore the mass of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (the *excess* reactant) that reacts is

$$\begin{aligned} \text{mass CaCl}_2 \cdot 2\text{H}_2\text{O} &= 0.194 \text{ g CaC}_2\text{O}_4 \cdot \text{H}_2\text{O} \times \frac{\text{mol CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}}{146.12 \text{ g CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} \\ &\quad \times \frac{1 \text{ mol CaCl}_2 \cdot 2\text{H}_2\text{O}}{1 \text{ mol CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}} \times \frac{147.02 \text{ g CaCl}_2 \cdot 2\text{H}_2\text{O}}{1 \text{ mol CaCl}_2 \cdot 2\text{H}_2\text{O}} \\ &= 0.195 \text{ g CaCl}_2 \cdot 2\text{H}_2\text{O} \text{ reacted} \end{aligned}$$

The mass of *excess* $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ is $0.293 \text{ g} - 0.195 \text{ g} = 0.098 \text{ g}$ *xs* $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$

Procedure Overview: In Part A a measured mass of a solid $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}/\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ salt mixture of unknown composition is added to water. The precipitate that forms is digested, filtered, and dried, and its mass measured. Observations from tests on the **supernatant** solution in Part B determine which salt in the mixture is the limiting reactant. An analysis of the data provides the determination of the percent composition of the salt mixture.

Two trials are recommended for this experiment. To hasten the analyses, measure the mass of duplicate unknown solid salt mixtures in *clean* 150- or 250-mL beakers and simultaneously follow the procedure for each. Label the beakers accordingly for Trial 1 and Trial 2 to avoid the intermixing of samples and solutions.

Obtain about 2–3 g of an unknown $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}/\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ salt mixture.

EXPERIMENTAL PROCEDURE

Supernatant: the clear solution that exists after the precipitate has settled



1. **Prepare the Salt Mixture.** a. *Mass of salt mixture.* Measure the mass ($\pm 0.001 \text{ g}$) of Beaker #1 and record on the Report Sheet for Trial 1. Transfer $\sim 1 \text{ g}$ of the salt mixture to the beaker, measure and record the combined mass. Repeat for Trial 2.

b. *Adjust pH of deionized water.* Fill a 400 mL beaker with deionized water. Test with pH paper . . . if the water is acidic, adjust it to basic with drops of 6 M NH_3 . If already basic to pH paper, then no addition of NH_3 is necessary.¹

c. *Mix deionized water and salt.* Add $\sim 150 \text{ mL}$ of the deionized water from Part A.1b to the salt mixture in Beaker #1. Stir the mixture with a stirring rod for 2–3 minutes and then allow the precipitate to settle. Leave the stirring rod in the beaker.

2. **Digest the Precipitate.** a. *Heat.* Cover the beaker with a watch glass and warm the solution on a hot plate (Figure 8.1) to a temperature not to exceed 75°C for 15 minutes. Periodically, stir the solution and in the meantime, proceed to Part A.3.

b. *Cool.* After 15 minutes, remove the heat and allow the precipitate to settle; the solution does *not* need to cool to room temperature.

c. *Wash water.* While the precipitate is settling, heat ($70\text{--}80^\circ\text{C}$) about 30 mL of deionized water for use as wash water in Part A.5.

A. Precipitation of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ from the Salt Mixture



¹Calcium oxalate does *not* precipitate in an acidic solution because of the formation of $\text{H}_2\text{C}_2\text{O}_4^-$, an ion that does not precipitate with Ca^{2+} .

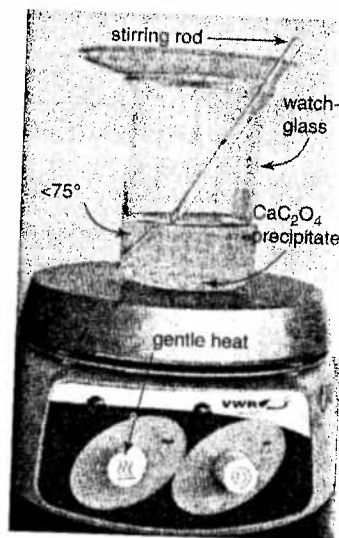


Figure 8.1 Warming and digesting the precipitate.



Rubber policeman: a spatula-like rubber tip attached to a stirring rod



- Set Up a Gravity (or Vacuum²) Filtering Apparatus.** Place your initials (in pencil) and Trial #1 on a piece of Whatman No. 42 or Fisherbrand Q2 filter paper,³ fold, and tear off its corner. Measure and record its mass (± 0.001 g). Seal the filter paper into the filter funnel with a small amount of deionized water. Discard the deionized water from the receiving flask. Have your instructor inspect your apparatus before continuing. Return to Part A.2b.
- Withdraw and Save Supernatant.** Once the precipitate has settled and the supernatant has cleared in Part A.2b, use a dropping pipet to withdraw enough supernatant to half-fill two 75-mm test tubes, labeled "1" and "2." Save for Part B.
- Filter the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ Precipitate.** While the remaining solution of the salt mixture from Part A.4 is still warm, quantitatively transfer the precipitate to the filter (Figure 8.2). Transfer any precipitate on the wall of the beaker to the filter with the aid of a **rubber policeman**; wash any remaining precipitate onto the filter with three or four 5-mL volumes of warm water (from Part A.2c).
- Dry and Measure the Amount of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ Precipitate.** Remove the filter paper and precipitate from the filter funnel. Air-dry the precipitate on the filter paper until the next laboratory period or dry in a $<110^\circ\text{C}$ constant temperature drying oven for at least 1 hour or overnight. Determine the combined mass (± 0.001 g) of the precipitate and filter paper. Record. Repeat for Trial 2.
- Formula of the Precipitate.** If the precipitate is air-dried, the precipitate is $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$; if oven-dried at $\geq 110^\circ\text{C}$, the precipitate is the anhydrous CaC_2O_4 . Enter the mass of the dried precipitate as *either* item A.6 (air-dried) or A.7 (oven-dried) on the Report Sheet.

²A vacuum filtering apparatus (Technique 11E) can also be used; the filtering procedure will be more rapid, but more precipitate may pass through the filter paper.

³Whatman No. 42 and Fisherbrand Q2 filter papers are both fine-porosity filter papers; a fine-porosity filter paper is used to reduce the amount of precipitate passing through the filter.



Figure 8.2 Gravity filtration is used to filter finely divided precipitates.

From the following two tests (Figure 8.3) you can determine the limiting reactant in the original salt mixture. Some cloudiness may appear in both tests, but one will show a definite formation of precipitate.

B. Determination of the Limiting Reactant



1. **Clarify the Supernatant.** Centrifuge the two collected supernatant samples from Part A.4.
2. **Test for Excess $C_2O_4^{2-}$.** Add 2 drops of the test reagent $0.5 M CaCl_2$ to the supernatant liquid in test tube 1. If a precipitate forms, the $C_2O_4^{2-}$ is *in excess* and Ca^{2+} is the limiting reactant in the original salt mixture.
3. **Test for Excess Ca^{2+} .** Add 2 drops of the test reagent $0.5 M K_2C_2O_4$ to the supernatant liquid in test tube 2. If a precipitate forms, the Ca^{2+} is *in excess* and $C_2O_4^{2-}$ is the limiting reactant in the original salt mixture.



An obvious formation of precipitate should appear in only one of the tests. Repeat for Trial 2.

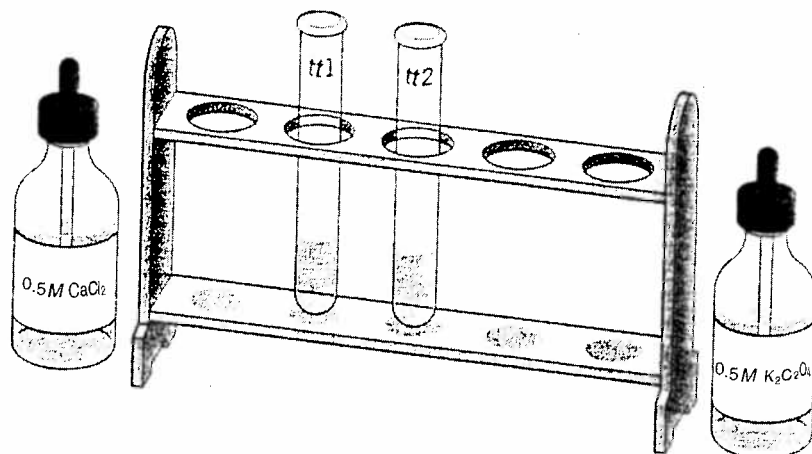


Figure 8.3 Testing for the excess (and the limiting) reactant.



4 *Disposal:* Dispose of the calcium oxalate, including the filter paper, in the "Waste Solids" container. Dispose of the waste solutions in the "Waste Liquids" container.



2 **CLEANUP:** Rinse each beaker with small portions of warm water and discard in the "Waste Liquids" container. Rinse twice with tap water and twice with deionized water and discard in the sink.

The Next Step

All reactions other than decomposition reactions have limiting reactants! From the combustion of fossil fuels to the many integrated chemical processes of biochemical reactions in living organisms, there is one reactant that limits the process. For examples, what is the limiting reactant in the combustion of gasoline in the cylinder of an engine; what is the limiting reactant in the eutrophication of a body of water; what is the limiting reactant in making bread rise; what is the limiting reactant in the precipitation of a salt, . . . ? Research the limiting reactant concept in upcoming experiments.

Experiment 8 *Prelaboratory Assignment*

Limiting Reactant

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

1. The limiting reactant is determined in this experiment.
 - a. What are the reactants (and their molar masses) in the experiment?

 - b. What is the product (and its molar mass) that is used for determining the limiting reactant?

 - c. How is the limiting reactant determined in the experiment?

2. Experimental Procedure, Part A.2. What is the procedure and purpose of “digesting the precipitate?”

3. Two special steps in the Experimental Procedure are incorporated to reduce the loss of the calcium oxalate precipitate. Identify the steps in the procedure and the reason for each step.

4. A 0.972-g sample of a $\text{CaCl}_2 \cdot 2\text{H}_2\text{O} / \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ solid salt mixture is dissolved in ~ 150 mL of deionized water, previously adjusted to a pH that is basic. The precipitate, after having been filtered and *air-dried*, has a mass of 0.375 g. The limiting reactant in the salt mixture was later determined to be $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$.
 - a. What is the percent by mass of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in the salt mixture?

b. How many grams of the excess reactant, $K_2C_2O_4 \cdot H_2O$, reacted in the mixture?

c. How many grams of the $K_2C_2O_4 \cdot H_2O$ in the salt mixture remain *unreacted*?

5. A 1.009-g mixture of the solid salts Na_2SO_4 (molar mass = 142.04 g/mol) and $Pb(NO_3)_2$ (molar mass = 331.20 g/mol) forms an aqueous solution with the precipitation of $PbSO_4$ (molar mass = 303.26 g/mol). The precipitate was filtered and dried, and its mass was determined to be 0.471 g. The limiting reactant was determined to be Na_2SO_4 .

a. Write the molecular form of the equation for the reaction.

b. Write the net ionic equation for the reaction.

c. How many moles and grams of Na_2SO_4 are in the reaction mixture?

d. How many moles and grams of $Pb(NO_3)_2$ reacted in the reaction mixture?

e. What is the percent by mass of each salt in the mixture?



Limiting Reactant

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

A. Precipitation of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ from the Salt Mixture

Unknown number _____

	<i>Trial 1</i>	<i>Trial 2</i>
1. Mass of beaker (g)	_____	_____
2. Mass of beaker and salt mixture (g)	_____	_____
3. Mass of salt mixture (g)	_____	_____
4. Mass of filter paper (g)	_____	_____
5. Mass of filter paper and $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (g)	_____	_____
6. Mass of air-dried $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (g)	_____	_____
or		
7. Mass of oven-dried CaC_2O_4 (g)	_____	_____

B. Determination of Limiting Reactant

1. Limiting reactant in salt mixture (write complete formula) _____
2. Excess reactant in salt mixture (write complete formula) _____

Data Analysis

1. Moles of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (or CaC_2O_4) precipitated (mol)	_____	_____
2. Moles of limiting reactant in salt mixture (mol)		
• formula of limiting hydrate _____	_____	_____
3. Mass of limiting reactant in salt mixture (g)		
• formula of limiting hydrate _____	_____	_____
4. Mass of excess reactant in salt mixture (g)		
• formula of excess hydrate _____	_____	_____
5. Percent limiting reactant in salt mixture (%)		
• formula of limiting hydrate _____	_____	_____
6. Percent excess reactant in salt mixture (%)		
• formula of excess hydrate _____	_____	_____
7. Mass of excess reactant that reacted (g)		
• formula of excess reactant _____	_____	_____
8. Mass of excess reactant, unreacted (g)	_____	_____

Show all calculations for Trial 1.

Laboratory Questions

Circle the questions that have been assigned.

1. Part A.2. If the step for digesting the precipitate were omitted, what would be the probable consequence of reporting the "percent limiting reactant" in the salt mixture? Explain.
2. Part A.3. A couple of drops of water were accidentally placed on the properly folded filter paper before its mass was measured. However, in Part A.6, the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate and the filter paper were dry. How does this sloppy technique affect the reported mass of the limiting reactant in the original salt mixture? Explain.
3. Part A.5. Because of the porosity of the filter paper some of the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate passes through the filter paper. Will the reported percent of the limiting reactant in the original salt mixture be reported too high or too low? Explain.
4. Part A.5. Excessive quantities of wash water are added to the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate. How does this affect the mass of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate reported in Part A.6?
5. Part A.6. The $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitate is not completely air-dried when its mass is determined. Will the reported mass of the limiting reactant in the original salt mixture be reported too high or too low? Explain.
6. Part A.6, 7. The drying oven, although thought (and assumed) to be set at 125°C , had an inside temperature of 84°C . How will this error affect the reported percent by mass of the limiting reactant in the salt mixture . . . too high, too low, or unaffected? Explain.