Qualitative Analysis

Adapted from "Inorganic Qualitative Analysis," Trinity Software.

For this experiment:

- Read through experiment carefully, along with reviewing the end of Chapter 18 and 5 (solubility rules!) in Kotz. (Or the solubility rules and K_{sp} section of another textbook).
- Complete the Prelab (last page) and get it stamped by the instructor ***before*** you begin to work on the computer simulations or the lab tests.
- Prepare your notebook ahead of time – create tables corresponding to all computer tests and all lab tests. Write out reactions (with phase labels!). Samples are in this handout.
- Complete the computer simulations. You may do these before or after doing any lab tests, however, you will not be given an unknown without showing the correct printouts from the computer simulations to the lab instructor. You must correctly identify **two** "1 cation" unknowns. The unknowns *must be different!* *Print* out the results for each unknown to turn in. Any student who does a nickel cation unknown on the computer will not be able to do a nickel cation unknown in lab! The computer simulation is available on the PCs in APH 102. You may download a 30-day trial at http://www.chem.wwu.edu/qual/.
- Complete all lab "tests" before doing any actual unknowns. You must do TWO unknown cations on the computer and 1 unknown cation and 1 unknown anion IN lab.

Turn in only the Data Report Sheet, the Stamped Prelab, the Computer Unknown Print–outs (2 cation unknowns), and the copies of your lab notebook pages.

In this experiment, you will carry out a number of reactions involving the following ions:

Cations:	Na^+	Mg^{2+}	Ni^{2+}	Cr^{3+}	Zn^{2+}	Ag^+	Pb^{2+}
Anions:	NO_3^-	Cl^-	I^-	SO_4^{2-}	OH^-		

The stock solutions of the cations are prepared from their **nitrate** salts ($NaNO_3$), $Ni(NO_3)_2$, etc.). The anion stock solutions are all **sodium** salts ($NaNO_3$, NaCl, NaI and Na_2SO_4). All nitrate and sodium salts are soluble in water. The other reagents used in this experiment are sodium hydroxide (6 M NaOH), ammonia (6 M NH_3), and the common strong acids (6 M HCl, 6 M HNO_3). Stock solutions of the cations and anions are all 0.1 M except the Cl^- solution which is 0.5 M (and slightly acidified with HNO_3).

The reactions you will perform involve the formation and dissolution of precipitates. These reactions can be used to **separate** and **identify** ions in an unknown substance. After studying these reactions, you will investigate the identification of some simple unknowns. The following reactions are to be investigated in the laboratory and may also be practiced using the computer program "Inorganic Qualitative Analysis" by Trinity Software – the software can be downloaded on your home PC computer (not Mac compatible, or accessed in APH 102 or at the library).

Reactions should be carried out carefully. Mixing of solutions is very important noting color changes precipitate formation etc. very carefully. Add reagents drop by drop. You may have to review writing and balancing equations, solubility rules, and net ionic reactions in order to complete this experiment.

Definitions for the following terms are important in understanding the laboratory. The ***Laboratory Handbook***, has techniques for using a centrifuge.

Centrifuge	supernatant liquid
Reagent	precipitate
Decant	stability sequence

Using the Computer simulation (at home or in computer lab)

1. There is an option of doing the computer simulations either by**
   a) doing the preliminary reactions and writing out the equations, or
   b) performing some reactions, and then using the computer and going back to the lab, and doing more reactions and so on. (**summer students should perform all reactions on the computer FIRST before performing reactions in the laboratory)

Try to establish a pattern in your procedures and **do all the lab reactions on the computer** as simulations before you do unknowns. **Practice** before attempting unknowns.

2. The **computer assignment** of identification of **two, "single cation" unknowns** with a printout of results for each must be completed prior to receiving an unknown for the lab.

Lab Tests

After viewing the reactions in the computer simulation, you will need to perform the various reactions in the lab. Pay close attention to colors and to the quality of the solids that form – observe the solid and note whether it is chunky, gelatinous, etc…

Unknowns

Each student must show the lab instructor a completed observation section and the appropriate net ionic reactions **in their notebook** and their computer printout from the simulation unknowns before they will be given a lab unknown. Do not write information on the lab report sheets, they are just sample formats.

There will be a single cation unknown, and a single anion unknown to identify in the laboratory.

I. Reactions of Cations with NaOH

All of the cations in this experiment except Na^+ form insoluble hydroxides. Thus a precipitate should appear when NaOH is added to any of the other cation stock solutions. Some of these hydroxide precipitates dissolve on adding excess NaOH and are said to be **AMPHOTERIC**. Hydroxides that do not dissolve in excess NaOH are called **BASIC** hydroxides.

Basic Hydroxides:	**$Mg(OH)_2(s)$**	**$Ni(OH)_2(s)$**	**$AgOH(s)$**
Amphoteric Hydroxides:	**$Cr(OH)_3$ (s)**	**$Zn(OH)_2(s)$**	**$Pb(OH)_2(s)$**
Species formed in xs NaOH:	$Cr(OH)_4^-$ (aq)	$Zn(OH)_4^{2-}$ (aq)	$Pb(OH)_4^{2-}$ (aq)

PROCEDURE: Measure ~1 mL portions (about 20 drops) of each of the seven cation solutions into separate test tubes. Add one drop NaOH solution to each, mix thoroughly (ask your instructor), note the reactions which occur, and fill in the data table below. Now add 10 additional drops NaOH to each test tube and mix thoroughly. With this excess, the amphoteric hydroxides will dissolve. Write balanced net ionic equations for all reactions.

Transfer these tables to your notebooks and make all comments and observations in your notebook!

Sample Data Table for Reactions with dropwise addition of NaOH

Cation	Precipitate?	Net Ionic Equation	Observations
Na^+	No	No Reaction	
Mg^{2+}	Yes	$Mg^{2+}{}_{(aq)} + 2\ OH^-{}_{(aq)} \rightarrow Mg(OH)_{2\ (s)}$	White, gelatinous ppt
Ni^{2+}			
Cr^{3+}			
Zn^{2+}			
Ag^+			
Pb^{2+}			

Sample Data Table for Reaction with *Excess* NaOH

Precipitate	Dissolve?	Net Ionic Equation	Observations
$Mg(OH)_2$ (s)	No	No Reaction	
$Ni(OH)_2$ (s)			
$Cr(OH)_3$ (s)	Yes	$Cr(OH)_{3\ (s)} + OH^-{}_{(aq)} \rightarrow [Cr(OH)_4]^-{}_{(aq)}$	
$Zn(OH)_2$ (s)			
AgOH (s)			
$Pb(OH)_2$ (s)			

II. Reactions of Cations with Ammonia

Ammonia is weakly basic. It provides sufficient hydroxide ions for formation of hydroxide precipitates, but not enough to dissolve the amphoteric hydroxides. However, ammonia does form soluble ammine complexes (complex ions) with nickel, zinc and silver cations: $Ni(NH_3)_4{}^{2+}$, $Zn(NH_3)_4{}^{2+}$ and $Ag(NH_3)_2{}^+$

PROCEDURE: Add 15 drops of NH_3 to 1 mL portions of each of the seven cation solutions, mix thoroughly, and note the reactions that occur. With this excess of NH_3, those cations which form ammine complexes will now be in solution. The other cations will precipitate as the hydroxide salts (except Na^+ which always forms soluble salts).

III. Anion Precipitation Reactions

You will investigate precipitation reactions with chloride, iodide and sulfate anions. It was previously stated that all nitrate salts are soluble in water. The Cl^- and I^- salts of the cations in this experiment are also soluble except for those of Ag^+ and Pb^{2+}. Thus if you mix the silver stock solution ($AgNO_3$) and the chloride stock solution (NaCl), precipitation of AgCl(s) will occur. $PbCl_2$ and PbI_2 differ from AgCl and AgI in that they are soluble in hot water. Sulfate salts have limited solubilities and concentration dependent. Some sulfate compounds are soluble, while others are insoluble, but may be dissolved when heated.

PROCEDURE: Combine 1 mL of silver nitrate to 1 mL portions of each stock solution of chloride, iodide and sulfate, mix well, and note the formation of precipitates. Heat each mixture containing a precipitate in boiling water, mix well, and note those precipitates which dissolve. Combine 1 mL portions of lead nitrate to 1 mL portions of each stock solution of chloride, iodide and sulfate, mix well, and note the formation of precipitates. **<u>Exception</u>: For the $Pb(NO_3)_2$ / NaI reaction, use 3 drops of each test solution and add 1 mL of H_2O.**

IV. Additional Reactions and Stability Sequences

In some cases, the individual tests studied thus far can be combined sequentially to obtain further useful information about an unknown sample. Below are some examples.

1. Ag^+ can be precipitated as AgCl(s) by addition of NaCl. If NH_3 is added to the sample, the AgCl(s) dissolves and $Ag(NH_3)_2^+$ forms:

$$\mathbf{AgCl(s)} + 2\ NH_3\ (aq) \rightarrow Ag(NH_3)_2^+ + Cl^-$$

2. If NaI is added to the solution formed above, AgI(s) forms:

$$Ag(NH_3)_2^+ + I^- \rightarrow \mathbf{AgI(s)} + 2\ NH_3\ (aq)$$

All of the products formed by Ag^+ can be ranked in order of increasing stability. This ranking is termed a ***stability sequence*** and is as follows:

$$\mathbf{AgOH(s)} < \mathbf{AgCl(s)} < Ag(NH_3)_2^+ < \mathbf{AgI(s)}$$

The analogous stability sequences for Pb^{2+}, Zn^{2+} and Ni^{2+} are:

$$\mathbf{PbCl_2(s)} < \mathbf{PbSO_4(s)} < \mathbf{PbI_2(s)} < \mathbf{Pb(OH)_2(s)} < Pb(OH)_4^{2-}\ (xs\ OH^-)$$

$$\mathbf{Zn(OH)_2(s)} < Zn(NH_3)_4^{2+} < Zn(OH)_4^{2-}\ (xs\ OH^-)$$

$$\mathbf{Ni(OH)_2(s)} < Ni(NH_3)_4^{2+} < Ni(OH)_2(s)\ (xs\ OH^-)$$

A number of reactions illustrating these sequences will be performed.

PROCEDURE: Record your observations and write net ionic equations for each of the following tests:

1. Combine 1 mL $AgNO_3$ and 1 mL NaCl. Heat to coagulate the solid, centrifuge, and discard the liquid. Add NH_3 to dissolve the solid. Then add NaI dropwise.
2. Precipitate some $PbCl_2$ (s), centrifuge, and discard the liquid. Add NaOH dropwise with stirring to the solid. Note any changes to the solid, then add excess NaOH.
3. Combine 1 mL Ni^{2+} stock solution and 1 mL NH_3. Mix and add 1 mL NaOH.

<u>Sample</u> Data Table - Observations and Equations

Procedure	**Observation**	**Net Ionic Equation**
Silver Stability Series: Combine Ag^+ and Cl^-	Precipitate	$Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)}$
Add NH_3		
Add I^-		
Lead Stability Series: Combine Pb^{2+} and Cl^-		
Add xs NaOH		
Nickel Stability Series: Combine Ni^{2+} and NH_3		
Add xs NaOH		

V. Analysis of Single Cation and Single Anion Unknowns

To illustrate the use of the reactions studied, suppose you have an unknown nitrate salt of one of the cations and are to identify that cation using any of the procedures in Sections I-IV. One possible set of tests is given below. **Study** the observations and verify that they lead to the stated conclusions.

Test Performed	Observation	Conclusion
NaCl added to a portion of unknown solution	No precipitate	Ag^{+}, Pb^{2+} absent
xs NaOH added to portion of unknown solution	Precipitate forms doesn't re-dissolve	Mg^{2+} or Ni^{2+} present
NH_3 added to a portion of unknown solution	No precipitate, blue complex	Unknown = Ni^{2+}

You will need to identify one unknown cation and one unknown anion. Using your notebook only (not this hand out), perform a series of chemical tests to determine the identities.

As you embark on identifying your unknown, think *carefully* about the order of reactions you follow. Remember: your goal is to eliminate possibilities to identify your unknown. Also, make notes an observations regarding what is in your test tube!

Possible cation unknowns include: Mg^{2+} Ni^{2+} Cr^{3+} Zn^{2+} Ag^{+} Pb^{2+} (note: if you identified a colored unknown on the computer, you may not take a colored unknown in lab!!)

Possible anion unknowns include: Cl^{-} I^{-} SO_4^{2-} OH^{-}

Chem 135 **Qualitative Analysis** **DATA REPORT SHEET**

Name ______________________________

PROCEDURE: Obtain a sample of an unknown cation and a sample of an unknown anion. Identify these ions using any of the tests from sections I-IV of the experiment. You may use your lab notebook, but not the handout so make sure all of the data and reactions are in the notebook. If the instructor catches you using your handout, you will get a zero for this part of the lab.

Do NOT discard or replace the unknown in the original rack or beaker. Keep it in your lab drawer in case there is a question about the result. Make sure you label it so the other person sharing your drawer knows which unknowns are yours.

Show all results in your notebook and record enough information here to justify your answer.

Cation Unknown **ID Number:** _______

Test	**Observation**	**Conclusion**
		Ans.

Anion Unknown **ID Number:** _______

Test	**Observation**	**Conclusion**
		Ans.

Prelab Questions **Qualitative Analysis** Name ___________________________

Stamp Here

1. Label the following salts as soluble (S) or insoluble (I) in 20.0°C water.

1. NaI	6. $Cr_2(SO_4)_3$
2. Na_2SO_4	7. $PbCl_2$
3. MgI_2	8. AgCl
4. $Zn(NO_3)_2$	9. AgI
5. $ZnSO_4$	10. $Mg(OH)_2$

2. $Pb(OH)_2$ (s) is amphoteric and forms the complex ion $Pb(OH)_4^{-2}$ ion with excess OH^- in solution.

Write balanced equation for the reaction of $Pb(OH)_2$ (s) with excess OH^- (aq)

____$Pb(OH)_{2\,(s)}$ + ____ $OH^{-1}{}_{(aq)}$ →

3. $Pb(OH)_{2\,(s)}$ is a solid that can be dissolved in the presence of an acid.

Write the balanced chemical equation for the reaction of $Pb(OH)_{2\,(s)}$ in the presence of acid ($H^{+1}{}_{(aq)}$)

_____$Pb(OH)_{2\,(s)}$ + _____$H^+{}_{(aq)}$ →

3. Based upon K_{sp} values given, circle the species that will form the most stable precipitate, AgI or AgCl?

AgI	AgCl
K_{sp} = 8.3×10^{-17}	K_{sp} = 1.8×10^{-10}

4. A solution contains Pb^{2+} and Zn^{2+} ions, with nitrate as the counter ion. Consult the solubility rules to answer the following questions.
   a. NaOH is added.

What precipitate (or precipitates) forms? ________________________________

Can you distinguish between the two metal ions using sodium hydroxide? Yes No.

   b. NaI is added.

What precipitate (or precipitates) forms? ________________________________

Can you distinguish between the two metal ions using sodium iodide? Yes No.

5. A solution contains an unknown anion. Another solution of magnesium nitrate is added to the unknown, and <u>no</u> precipitate forms.

What anion can it be? Circle all that apply Cl^- I^- SO_4^{2-} OH^-

What anion can't it be? Circle all that apply Cl^- I^- SO_4^{2-} OH^-