Experiment 11. Qualitative Analysis of Ions
Pre Laboratory Assignment

Name: ___________________________________

Lab Section________

1. The elements in group IA belong to the alkali metal family. You have been given aqueous test solutions of 1 M sulfuric acid and 1 M hydrochloric acid. Explain how you would use these test solutions to determine if an unknown aqueous solution contained Ag^{+}, Ba^{+2}, or Na^{+} ions. Use complete sentences and clear explanations. In other words, explain the tests you would perform, the expected outcomes, and how the results would allow you to determine the presence or absence of the particular cations.

2. The ions A^{-}, B^{-}, and C^{-} all form colorless aqueous solutions. Their molecular counterparts, however, form brilliantly colored solutions, where A_2 is green, B_2 is blue, and C_2 is red.

When a student mixes the anions C^{-} and A^{-} with a solution of B_2, the following results were obtained:

i) $B_2 + 2\ C^{-} \rightarrow$ red solution

ii) $B_2 + 2\ A^{-} \rightarrow$ blue solution

a) Rank A_2, B_2, and C_2 in order of increasing oxidizing strength.

b) Would the following reaction occur? Explain why or why not.

$$A_2 + 2\ C^{-} \rightarrow C_2 + 2\ A^{-}$$

c) Devise a single–step test that one could use to analyze an unknown solution containing one of the three anions: A^{-}, B^{-}, or C^{-}. Explain how and why this would work.

EXPT 11. QUALITATIVE ANALYSIS - AN INVESTIGATION OF SOME GROUP IIA AND VIIA ELEMENTS

I. Discussion

Elements in the same column of the periodic table (called a group or family) have similar chemical properties. Further, we would predict that the properties within a group would vary continuously from the top to the bottom of a column. In this experiment, we will use this gradual variation in properties to develop a scheme to identify what chemicals are present in an unknown solution.

The families we will be investigating are the alkaline earths (Group IIA) and the halogens (Group VIIA). We will establish which of the "safe" alkaline earths (beryllium compounds are extremely toxic and radium is radioactive, as the name implies) have the greatest tendency to precipitate with a series of anions, A, in aqueous solution. [No, don't bother looking for the element A in the Periodic Table; here, A represents the carbonate ion (CO_3^{2-}), chromate ion (CrO_4^{2-}), oxalate ion ($C_2O_4^{2-}$), or the sulfate ion (SO_4^{2-}).] You will find that the elements at each end of the column IIA represent two extremes; one hardly forms precipitates at all, and the other will precipitate with just about any of these anions.

Similarly, we will develop a procedure to identify a halogen anion present. The property we will use to distinguish between Cl^-, Br^-, and I^- is called oxidation (you will learn a lot more about this in chemistry 1B). A compound is oxidized when it loses electrons to another compound. The other compound taking the electrons is called an oxidizing agent. Oxidation is always accompanied by a reduction which means the gain of electrons. In explanation, the oxidizing agent is reduced when it gains electrons from the compound being oxidized. It turns out that three halogen molecules, Cl_2, Br_2, and I_2, are fairly effective oxidizing agents (able to take electrons from other compounds). In fact, commercial liquid bleaches are solutions of Cl_2 in water. These solutions use the tendency of chlorine to remove electrons from stains to make them water soluble.

$$Cl_2 + \text{stain} \rightarrow 2\ Cl^- + \text{stain}^{2+}$$

We will investigate a series of reactions of the general form

$$X_2 + 2\ Y^-(aq) \rightarrow 2\ X^-(aq) + Y_2$$

to determine which of chlorine (Cl_2), bromine (Br_2), or iodine (I_2) is the strongest oxidizing agent (able to best take electrons from another compound). When this is determined, the superior oxidizing agent can be added to the unknown solution to convert the halide ion that was present to the neutral, diatomic (halogen) form. At this point, a little clarification may by in order; we will be dealing with six entities:

	Halides		Halogens
Cl^-	chloride ion	Cl_2	chlorine
Br^-	bromide ion	Br_2	bromine
I^-	iodide ion	I_2	iodine

Your unknown will contain one of the halide anions (Cl^-, Br^-, or I^-) and one of the alkaline earth metal cations. Your goal will be to find out which anion and cation are present. The identity of the halide ion will be easy to determine because of two facts: neutral compounds (like Cl_2, Br_2, and I_2) tend to be more soluble in organic solvents than in water (the opposite is true for ionic substances), and the neutral halogens have a characteristic color in the organic solvent (cyclohexane, C_6H_{12}).

II. Experimental Procedure

1. Alkaline earths and their relative solubilities.

Add about 1 mL of each alkaline earth metal nitrate solution to separate small test tubes. Add to each solution 1 mL of 1 M Na_2CO_3. Shake gently to insure thorough mixing and record the results in Table 1. If a precipitate forms, note the characteristics of the precipitate (color, size of particles, heavy or light precipitate, etc.). Repeat the experiment (after washing the test tubes) with 0.25 M $(NH_4)_2C_2O_4$, with 1 M K_2CrO_4, and with 1 M Na_2SO_4, and record your results in Table 1.

2. The halogens and their relative oxidizing power.

a) **Color test** - Place 1 mL bromine-saturated water in a clean, small test tube with about 1 mL C_6H_{12}, cork the test tube and shake vigorously. Note the color in the upper (C_6H_{12}) layer. This is the characteristic color of Br_2 in C_6H_{12}. Repeat the experiment, using the saturated Cl_2 solution with C_6H_{12} and again using the saturated I_2 solution with C_6H_{12}. In each case, note the color of the upper layer.

b) **Oxidizing ability** - Place 1 mL each of bromine water and C_6H_{12} in two clean test tubes. To one, add 1 mL 0.1 M NaCl solution and to the other add 1 mL 0.1 M NaI solution. Stopper each solution and shake well. Note the color in the upper (C_6H_{12}) layer. Which halogen is present? Did a reaction occur or not? Record the results in Table 2. If a reaction did occur, Br_2 is a stronger oxidizing agent than the halogen that was produced; if not, it is weaker than the halogen that would have been produced had a reaction occurred. Repeat the tests using Cl_2 water with NaBr and NaI. Finally, use I_2 water with NaCl and NaBr solutions to complete Table 2. Use C_6H_{12} to extract and identify the halogen in each case.

3. Identification of the unknown

Before you try to determine the identity of the ions in your unknown solution (which you should obtain from your laboratory instructor), outline procedures for doing each determination. The most efficient procedure would involve only two tests to determine the identity of the cation and only one test to determine which anion you have. Write the procedure in the form of a flow chart and submit the procedure to your instructor for approval. If it is approved, note the actual results you obtained when you followed your procedure. Finally, record the contents of your unknown solution. Do not forget to record your unknown number.

III. Data and Observations: *(Turn in only this and the following pages.)*

Name: ________________________________

Section: __________

A. Solubility results

TABLE 1.

	1M Na_2CO_3	1M K_2CrO_4	0.25 M $(NH_4)_2C_2O_4$	1M Na_2SO_4
$Ba(NO_3)_2$				
$Ca(NO_3)_2$				
$Mg(NO_3)_2$				
$Sr(NO_3)_2$				

Arrange the alkaline earth cations in order of **decreasing** solubility.

___________ ___________ ___________ ___________

How does your ordered list compare to the periodic table?

B. Oxidation results

1. Color of the halogen in C_6H_{12}

	Br_2	Cl_2	I_2
Color	__________	__________	__________

2. Reactivity

TABLE 2. State the final color in the upper (C_6H_{12}) layer and the identity of the halogen present. State if a reaction did or did not occur.

	Br_2	Cl_2	I_2
Br^-	NR		
Cl^-		NR	
I^-			NR

Instructor date and initial: ______________________________

C. The Unknown

1. Procedure for identifying a Group IIA cation.

(a) Write a net ionic equation for all ppt. reactions observed in part A, Table 1.

(b) Devise a flowchart that describes the procedure for identifying each of the cations using two precipitation reactions. To begin, assume that you have a solution where all four cations (Ca^{2+}, Ba^{2+}, Mg^{2+}, & Sr^{2+}) are present. Next chose a solution containing one of the four anions (SO_4^{2-}, CO_3^{2-}, $C_2O_4^{2-}$, & CrO_4^{2-}) that when added divides the four cations into two groups of two cations: one group that forms a ppt and one that does not. Once you've done this, further divide the two groups of two into individual cations by choosing another anion of the four that separates each of the two cations in each group into individual cations by precipitation or no reaction.

Ca^{2+} Ba^{2+} Mg^{2+} Sr^{2+}

Instructor date and initial: ______________________________

2. Procedure for identifying a Group VIIA anion.

Devise a one–step test that will allow you to identify a solution containing one of the three halides Cl^-, Br^-, or I^- by adding a solution containing one of the three halogens.

3. Your unknown results Unknown number ___________________

Cation test observations, and net ionic equations for positive tests:

Anion test observations, and net ionic equations for positive tests:

Cation present ________ Anion present ________