

Chemical Properties of the Alkaline Earths & the Halogens

Cautions

The majority of the compounds in today's lab are harmful so be careful not to come in to contact with any of the compounds.

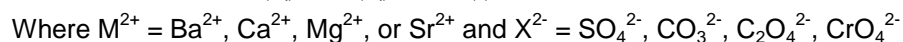
Purpose

To use observational skills and knowledge of simple chemical reactions to determine the identity of an unknown salt.

Introduction

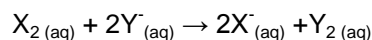
The periodic table organizes elements in order of increasing atomic number such that elements with similar properties are located beneath each other. The elements in a given column are referred to as a family or group. The physical and chemical properties in a given group will gradually change from one element to a next. By observing trends in properties, the elements can be arranged in order on the periodic table. In this experiment you will study the properties of the elements in two families: Alkaline Earth Metals and Halogens.

The alkaline earth metals, barium, beryllium, calcium, magnesium, radium, and strontium, are a moderately reactive family of elements. Since beryllium compounds are rarely encountered and usually poisonous and radium is radioactive, these will not be included in this experiment. The alkaline earth metals tend to lose electrons in reactions to form M^{2+} cations (Mg^{2+} , Ca^{2+} , etc...). When solutions of M^{2+} are mixed with solutions containing X^{2-} anions (CO_3^{2-} , SO_4^{2-} , $C_2O_4^{2-}$ (oxalate) etc...), salts of the alkaline earths will precipitate if the compound MX is insoluble.



The solubility of the alkaline earth salts generally show a smooth trend consistent with the order of the periodic table, e.g. the solubility of sulfate salts will gradually change from magnesium through barium. Similar trends exist for other anions in this experiment. By evaluating these trends, you will be able to confirm the order of the alkaline earth metals in the periodic table.

The halogens are relatively reactive and include astatine, iodine, bromine, chlorine, and fluorine. We will not study fluorine in this experiment since it is too reactive to be used safely in our laboratory. Astatine will also be excluded since it is radioactive. The stable halogens are diatomic elements, existing in nature as F_2 , Cl_2 , Br_2 , and I_2 . Unlike the alkaline earth metals, the halogens tend to gain electrons forming halide anions (F^- , Cl^- , Br^- , and I^-). Because of this property, the halogens are oxidizing agents which tend to remove electrons from other species and are easily reduced. An interesting and simple of oxidation may occur when a solution containing a halogen is mixed with a solution containing a halide ion.



The reaction will occur if X_2 is a better oxidizing reagent than Y_2 , since X_2 can produce Y_2 by removing electrons from the Y^- ions. However if Y_2 is a better oxidizing agent than X_2 the reaction will not occur.

In the second part of the experiment, you will mix solutions of halogens and halide ions to determine the relative oxidizing strengths of the halogens. A periodic trend will be observed. By observing colors one is able to determine whether a reaction occurred. In water, and particularly in some organic solvents, the halogens have characteristic colors. The halide ions are colorless in water and insoluble in organic solvents. You will observe the characteristic colors of each neutral halogen molecule displays in hexane.

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If you combine an aqueous solution of $\text{Br}_2(\text{aq})$ and hexane, the hexane layer will float above the water layer since hexane is not soluble in water and less dense. After mixing the two solutions, most of the non-polar bromine will be transferred from the polar, aqueous layer to the non-polar, hexane layer. The Br_2 dissolved in the hexane layer will begin to appear as a deep orange color and the water layer will decrease in the characteristic color of bromine. *At this point no chemical reaction has occurred.* If a different halide is added to the same test tube with the bromine and hexane and the solutions are vigorously mixed, a chemical reaction may occur. Remember that all halide ions are colorless in water and insoluble in hexane, if no change in color in the hexane layer is observed then bromine is still present (no reaction has occurred). If, on the other hand, the orange color of bromine is replaced by the characteristic color of another halogen then a reaction has occurred. The bromine has been reduced to the bromide ion and the halide ion has been oxidized to the elemental halogen. *You will learn a chemical reaction has occurred by observing changes in the color within the hexane layer. You will determine what elemental halogen is present by observing the specific colors.*

One difficulty involves the names of the ions and the elements. You must learn both the names and properties of the halogen and the halide ions since these are different chemical species even though their names appear similar. Some of the properties we will use today are:

HALOGENS (element)	HALIDES (ions)
Diatomic Molecules	Ions in solution exist only in H_2O
Oxidizing Agents	Not an Oxidizing Agent
Odor	Odorless
Only slightly soluble in H_2O	Insoluble in hexane
Very Soluble in Hexane	
Distinctive color in Hexane	Colorless in water

Procedure

Note: 20 drops of a solution is approximately 1 mL.

A. Determination of the Relative solubility of the Alkaline Earth Salts

- A1. Add 1 mL of 1M H_2SO_4 to 4 small test tubes.
- A2. Add 1 mL of 0.1M solution of the nitrate salt of barium to the first tube.
- A3. Add 1 mL of the calcium, magnesium, and strontium salt solutions.
- A4. Mix solutions well in each test tube and record your observations on Table I then rinse out the test tubes.
- A5. Repeat steps a-d using 1mL of 1 M Na_2CO_3 instead of H_2SO_4 .
- A6. Repeat steps a-d using 1 mL of 0.25 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$.
- A7. Repeat steps a-d using 1 mL of 1 M K_2CrO_4 and 1 mL of 1 M Acetic Acid.

B. Determination of the Characteristic Halogen Colors.

- B1. Add ~2 mL of bromine saturated water to a small test tube.
- B2. Add 2 mL of hexane to the bromine saturated water.

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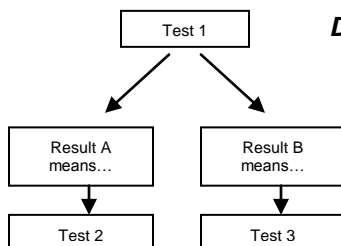


- B3. In the hood, mix each test tube well using a pipette until the color of the top hexane layer is unchanging. *Do not breath the vapors and do not use your finger to stopper the tube halogens cause severe chemical burns.*
- B4. Continue mixing till the color intensities in both layers are constant
- B5. Record you observations on Table II
- B6. Repeat steps a-e for the chlorine.
- B7. Repeat steps a-e for the iodine.



C. Determination of the Characteristic Halogen Relative Oxidizing Power

- C1. Obtain two small test tubes.
- C2. Add ~2 mL of bromine water and 1 mL of hexane to each.
- C3. Add 2 mL of 0.1M NaCl to one and 2 mL of 0.1M NaI to the second test tube.
- C4. Mix each test tube well using a pipette.
- C5. Continue mixing until the colors in both layers are no longer changing
- C6. Record you observations on Table III
- Note the color of the aqueous and hexane layer
 - Note whether a reaction occurred in the test tube.
 - If the color of the hexane layer is not the color of bromine (compare to Table II), then a chemical reaction has occurred.
- C7. Repeat C1-C6 for the aqueous chlorine solution while using NaBr and NaI.
- C8. Repeat C1-C6 for the aqueous iodine solution while using NaCl and NaBr.



D. Development of a Flow Chart for Determining an Unknown

- D1. Develop 2 flow charts – 1 for determining the Alkaline Earth and 1 for the Halide
- Use the solubility Data from Table I to determine the alkaline earth cation.
 - Use the oxidizing power data from Table III to determine the halide ion.
- D2. Get Instructor approval before going to E.

E. Determination of an Unknown

- E1. Obtain an unknown salt solution. Each student must perform the unknown identification separate from their partner and on a **different** unknown solution. Your unknown will be a salt of the form MX_2 where M^{2+} is an alkaline earth metal cation and X^- is a halide anion.
- E2. Using your flow charts and the other data collected in today's lab determine your unknown.
- E3. Record all your observations and detail your tests performed on the provided page.

Disposal

- All solutions containing **Barium** or **Chromate** must be placed in the appropriate **waste container**.
- All other **aqueous** solutions may be poured down the sink with an excess of water.
- All **hexane** waste must be placed in the **Organic Waste** container.

Clean-Up

- Wash all glassware with soap then rinse 3 times with tap water, and once with deionized water.

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Data Sheet

Name: _____

Determination of the Relative solubility of the Alkaline Earth Salts

Indicate whether a precipitate formed by placing a **P** in the box along with the **color** formed. If no precipitate was formed place an **S** in the appropriate box.

TABLE I	Chemical Formula	1 M H ₂ SO ₄	1 M Na ₂ CO ₃	0.25 M (NH ₄) ₂ C ₂ O ₄	1 M K ₂ CrO ₄
Barium Nitrate					
Calcium Nitrate					
Magnesium Nitrate					
Strontium Nitrate					

Determination of the Characteristic Colors of Halogens

TABLE II	Br ₂	Cl ₂	I ₂
Dissolved in Hexane			
Dissolved in Water			

Determination of the Relative Oxidizing Power of the Halogens and Halides

Circle "R" if a chemical reaction occurred or "NR" if no reaction occurred. In the top box, record the color observed in the hexane layer after adding and mixing the sodium halide water. In the bottom box, record the color of the water layer.

TABLE III	Br ⁻	Cl ⁻	I ⁻
Br ₂		R	R
		NR	NR
Cl ₂	R		R
	NR		NR
I ₂	R	R	
	NR	NR	

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Development of a Flow Chart for Determining an Unknown

A flow chart shows the sequence of activities, steps, and decision points that occur in a particular, discrete process. See p. 3 for an example. Design a process to identify the alkaline earth cation and halide anion present in your unknown salt. In addition to listing the tests you will use in the order you choose, include your expected results for each step and how you will proceed through the identification. You may use boxes and arrows or a simple outline format. *****NOTE - Never mix the oxalate reagent with sulfuric acid, because the sulfuric acid will convert the oxalate to carbon dioxide and will not allow for a successful test of calcium.*****

Both Flow Charts need to be approved by your Instructor before attempting to identify your Unknown

Flowchart for Identification of an Alkaline Earth Metal Cation:

Flowchart for Identification of a Halide Anion:

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Determination of an Unknown Salt

Include a COMPLETE description of procedures, observations, tests and other reasoning that you performed in determining the identity of your unknown. Feel free to reference your flow charts if it is helpful. This must be done for complete credit.

Results

Unknown # _____ Cation present _____ Anion present _____

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Pre-lab Assignment

Name: _____

1. For the table below, write the molecular formula for the species formed when the cation combines with the anion in the top cell and the name of the species in the bottom cell.

	I ⁻	NO ₃ ⁻	SO ₄ ²⁻	Cl ⁻	CO ₃ ²⁻	C ₂ O ₄ ²⁻	CrO ₄ ²⁻
Ba ²⁺							
Sr ²⁺							
Ca ²⁺							
Mg ²⁺							

2. A, B and C are monoatomic substances that can all act as oxidizing agents. In solution A is green, B is yellow and C is red. When A, B, and C are reduced A⁻, B⁻, and C⁻ are produced A⁻, B⁻, and C⁻ are also colorless ions in solution. When solution of A is mixed with a solution containing B⁻ ions the color changes from green to yellow.

i. Write a balanced chemical equation for the chemical reaction:

ii. Which species is oxidized? _____

iii. Which species is reduced? _____

When solution A is mixed with a solution containing C⁻ ions, the color remains green

iv. Is A a better or stronger oxidizing agent than C? _____

v. Is A a better or stronger oxidizing agent than B? _____