

EXPERIMENT C6: QUALITATIVE ANALYSIS OF CATIONS

Learning Outcomes

Upon completion of this lab, the student will be able to:

- 1) Analyze a given sample of an ionic compound and identify which of the following cations are present: silver, lead (II), bismuth (III), iron (III), manganese (II), aluminum, chromium (III), barium, strontium, calcium, nickel (II), copper (II), magnesium, zinc

Introduction

The cations being tested in the qualitative analysis scheme are organized into groups labeled A through D (Table 1)

Group	Cations
A	Ag ⁺ , Pb ²⁺
B	Bi ³⁺ , Fe ³⁺ , Mn ²⁺ , Al ³⁺ , Cr ³⁺
C	Ba ²⁺ , Sr ²⁺ , Ca ²⁺
D	Ni ²⁺ , Cu ²⁺ , Mg ²⁺ , Zn ²⁺

TABLE 1

This grouping of ions was arrived at based on the differences in the solubility of these ions.

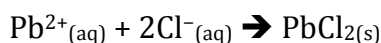
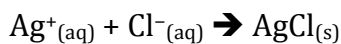
GROUP A: Ag⁺, Pb²⁺

The K_{SP} of the chloride salts of the two ions in this group is given in Table 2.

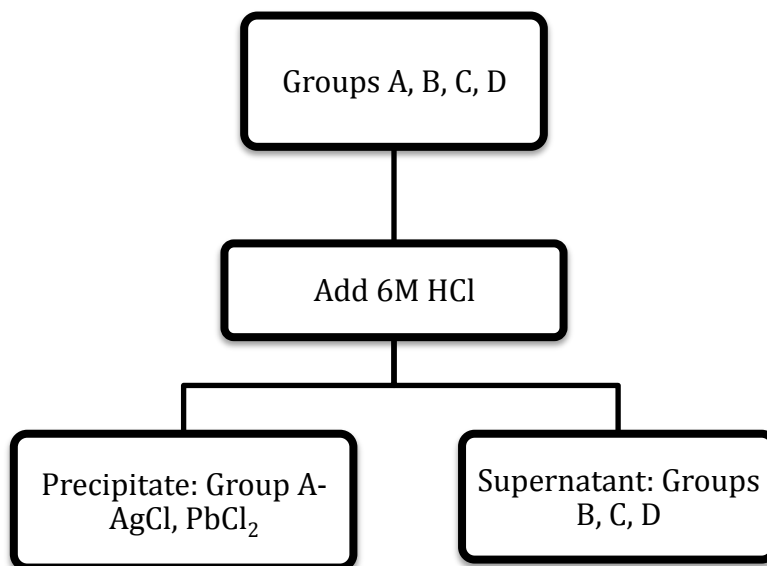
Salt	K _{SP}
AgCl	1.7 × 10 ⁻¹⁰
PbCl ₂	1.6 × 10 ⁻⁵

TABLE 2

The chloride salts of all the other cations that will be analyzed in this experiment are highly soluble. Therefore, addition of aqueous HCl will result in a precipitate containing chlorides of the Group A cations.



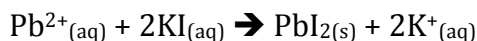
The chlorides of all other cations will remain in solution. When this mixture is centrifuged: the precipitate will contain the group A cations and the supernatant will contain the cations from the other groups.



Of the two chloride precipitates, PbCl_2 is soluble in hot water and AgCl is soluble in aqueous ammonia.

Test for Pb^{2+}

When the chloride precipitates of the Group A cations are mixed with hot water, PbCl_2 will dissolve. This is separated from AgCl by centrifuging the mixture. The PbCl_2 in the supernatant can be confirmed by reacting the supernatant with a solution of KI , which results in a yellow precipitate of PbI_2 .



Test for Ag^+

The AgCl precipitate dissolves in aqueous ammonia due to the formation of a complex ion.



Addition of HNO_3 results in the decomposition of the silver ammonium complex. The free silver ions combine with the chloride ions from the above reaction to form the AgCl precipitate again.

GROUP B: (Bi³⁺, Fe³⁺, Mn²⁺, Al³⁺, Cr³⁺)

The hydroxides of the cations in this group are almost completely insoluble. The K_{SP} of the hydroxide salts are given in Table 3.

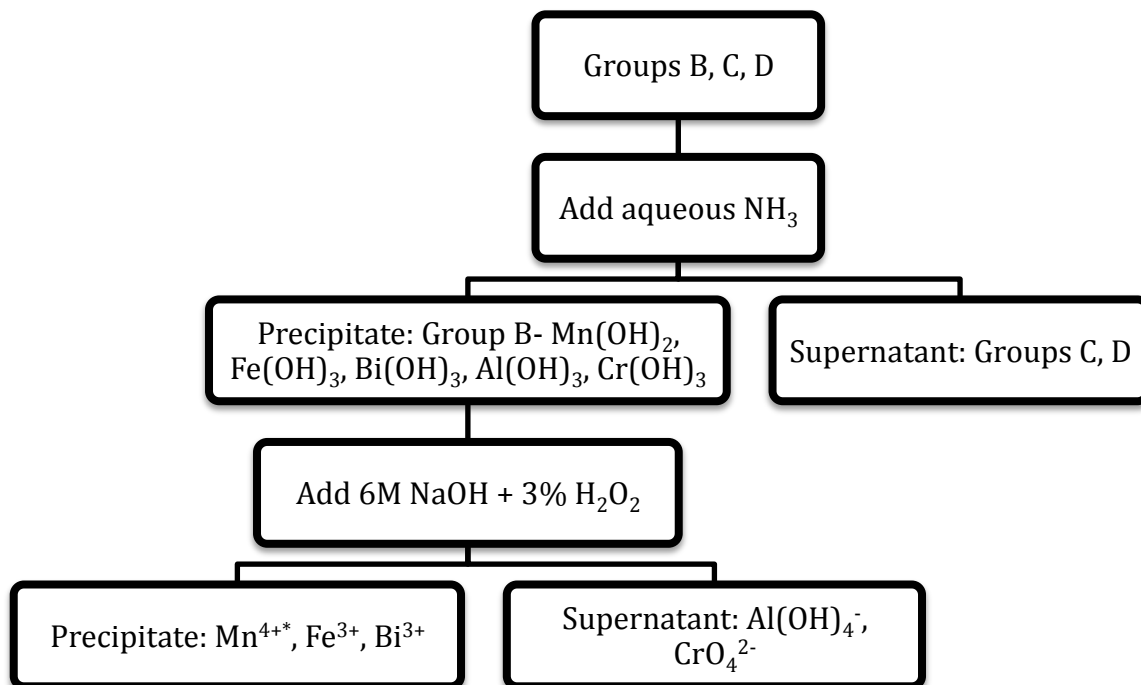
Salt	K_{SP}
Mn(OH) ₂	4.5×10^{-14}
Fe(OH) ₃	1.0×10^{-36}
Bi(OH) ₃	Insoluble
Al(OH) ₃	3.7×10^{-15}
Cr(OH) ₃	6.7×10^{-31}

TABLE 3

The hydroxide salts of the Group C cations are far more soluble. Although, the hydroxide salts of the Group D cations are also quite insoluble, the distinction between the hydroxide salts of Group B cations vs. Group D cations is that, the hydroxide salts of the Group D cations are soluble in aqueous ammonia, whereas those of the Group B cations are not.

Recall that the original mixture of cations was treated with HCl. This would not only lower the pH but also result in the precipitation of the Group A cations as chlorides. After separating the Group A cations by centrifugation, the supernatant solution containing all the rest of the cations should be treated with aqueous NH₃. This would raise the pH (i.e. increase the [OH⁻]) and precipitate the Group B cations. The supernatant containing the cations from Groups C and D can be obtained by centrifugation.

Of the five cations in this group, the hydroxides of Al³⁺ and Cr³⁺ are both soluble in an alkali. So, this group of cations is subdivided into two categories: B1, which contains Mn²⁺, Fe³⁺, and Bi³⁺ and B2, which contains Al³⁺ and Cr³⁺.

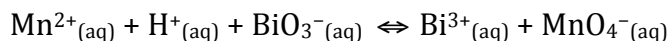


*NOTE: The Mn^{2+} is oxidized to Mn^{4+} by the H_2O_2

The B1 cations are all soluble in HCl. The precipitate containing the B1 cations is dissolved in hot HCl solution. This solution is then tested for each of the three B1 cations.

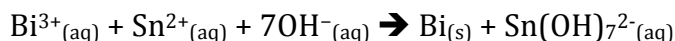
Test for Mn^{2+}

In an acidic environment the Mn^{4+} is reduced to Mn^{2+} using H_2O_2 . The Mn^{2+} is converted to MnO_4^- using bismuthate, BiO_3^- . The formation of a purple colored solution (from the MnO_4^-) confirms the presence of Mn^{2+} . The bismuthate is a strong oxidizing agent and causes a vigorous reaction and must therefore be added in small portions.



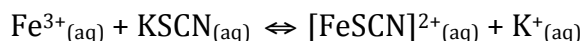
Test for Bi^{3+}

Bi^{3+} has a higher reduction potential ($E^\circ_{\text{RED}} = 0.286 \text{ V}$) than Sn^{2+} ($E^\circ_{\text{RED}} = -0.136 \text{ V}$). Therefore Sn^{2+} reduces Bi^{3+} . The reaction happens under basic conditions. The formation of a black precipitate, which is the element Bi, confirms the presence of Bi^{3+} .

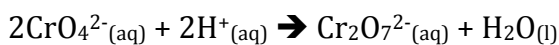
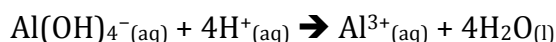


Test for Fe³⁺

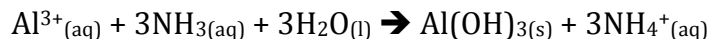
Fe³⁺ forms a complex with thiocyanate, SCN⁻. Addition of potassium thiocyanate to Fe³⁺ produces a reddish-brown color due to the formation of this complex. The formation of the reddish-brown color confirms the presence of Fe³⁺.

**B2 cations**

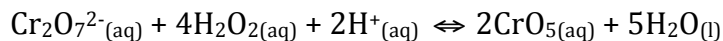
Treatment with 6 M NaOH and 3% H₂O₂ converts the B2 cations to Al(OH)₄⁻ and CrO₄²⁻. Under acidic conditions the following reactions occur.

**Test for Al³⁺**

The solution containing the Al³⁺ is treated with aqueous NH₃. This results in the formation of a white aluminum hydroxide precipitate. The dye aluminon when added to the precipitate, changes the color of the precipitate. The appearance of a red color confirms the presence of Al³⁺.

**Test for Cr³⁺**

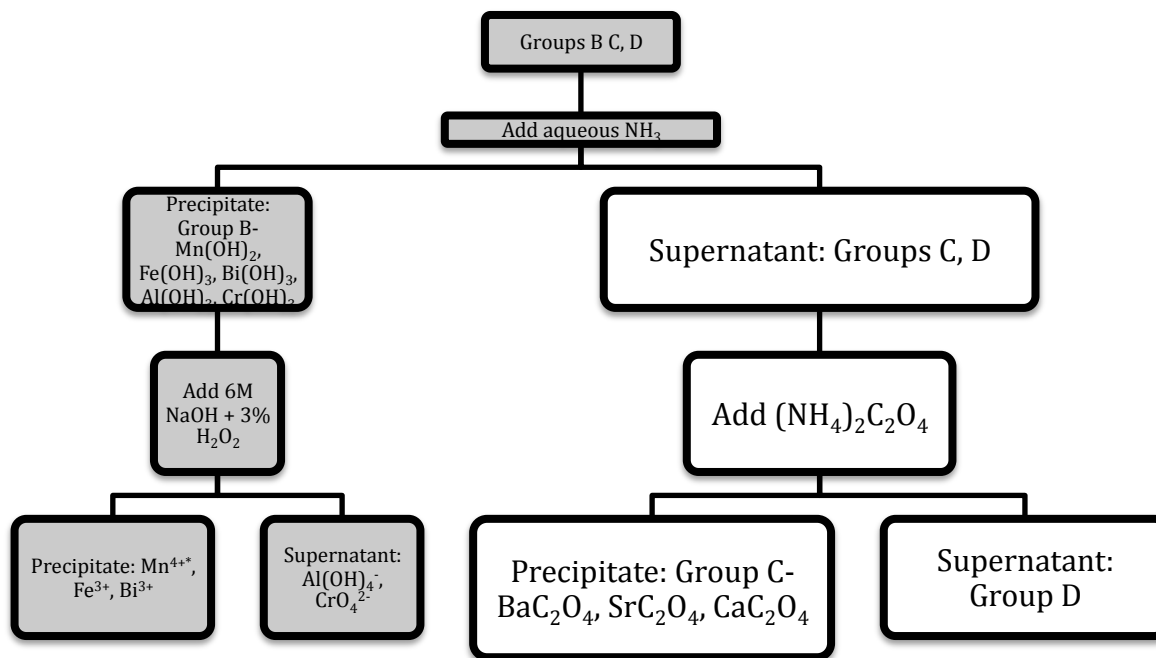
Any Cr³⁺ present in the test solution is found as Cr₂O₇²⁻ at this point. In an acidic medium the dichromate is oxidized by H₂O₂ to peroxochromate, CrO₅. The appearance of a dark blue colored CrO₅ confirms the presence of Cr³⁺. However, the CrO₅ is unstable and decomposes quickly. This causes the color to disappear.

**GROUP C: (Ba²⁺, Sr²⁺, Ca²⁺)**

The similarity between cations of Groups C and D is that they both form insoluble salts with oxalate anion. However, the Group D oxalate salts are soluble in aqueous ammonia whereas the Group C oxalate salts are insoluble in aqueous ammonia.

Note that after separating the Group B cations, the solution is already in a medium containing aqueous ammonia and at a high pH. Therefore, when ammonium oxalate

is added to this mixture containing dissolved ions of Groups C and D, the Group C oxalate ions precipitate.



The oxalate in the precipitate is removed by heating the precipitate with HNO_3 in a crucible. The oxalate is converted to $\text{CO}_2(\text{g})$. The precipitate is then dissolved in HCl . The three cations in this group will be analyzed by performing the flame test with this solution. If the unknown mixture is known to contain more than one of Ba^{2+} , Sr^{2+} , or Ca^{2+} , then they must be separated first prior to conducting the flame test.

Test for Ba^{2+} , Sr^{2+} , Ca^{2+}

A flame test is performed to analyze the presence of Ba^{2+} , Sr^{2+} , or Ca^{2+} .

- A yellow-green light confirms the presence of Ba^{2+} .
- A crimson color confirms the presence of Sr^{2+} .
- A brick-red color confirms the presence of Ca^{2+}

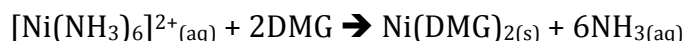
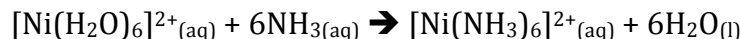
GROUP D: (Ni^{2+} , Cu^{2+} , Zn^{2+} , Mg^{2+})

The supernatant solution following the removal of the Group C cations, contains the oxalate salts of the Group D cations that have been dissolved in aqueous ammonia. The oxalate as well as the ammonia will first be removed by heating the sample to

dryness. Oxalates are removed as CO₂ gas and ammonium ions are removed as N₂O gas. The residue at this stage is dissolved in HCl which results in a solution that may be bluish green in color and will be used for analysis of the Group D cations.

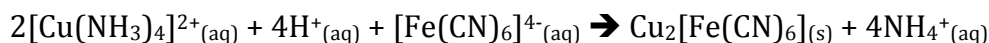
Test for Ni²⁺

Under basic conditions, Ni²⁺ forms a cherry red color precipitate with dimethylglyoxime (DMG). The formation of the red color precipitate confirms the presence of Ni²⁺.



Test for Cu²⁺

Under slightly acidic condition, Cu²⁺ forms a maroon precipitate with hexacyanoferrate. The formation of the maroon precipitate confirms the presence of Cu²⁺.

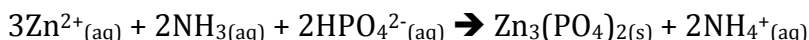


Removal of Ni²⁺ and Cu²⁺

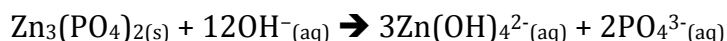
Sulfides of Ni²⁺ and Cu²⁺ are black insoluble solids. These precipitates are centrifuged and removed prior to the analysis for Mg²⁺ and Zn²⁺

Test for Mg²⁺

The test solution containing Mg²⁺ and/or Zn²⁺ is treated with aqueous NH₃ and Na₂HPO₄. Zn²⁺, if present precipitates as Zn₃(PO₄)₂ and Mg²⁺, if present precipitates as MgNH₄PO₄.

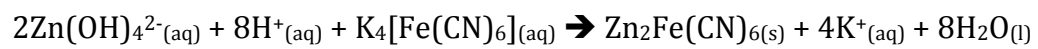


The precipitates are dissolved in NaOH; only the Zn₃(PO₄)₂ dissolves. Presence of a white precipitate after addition of NaOH confirms the presence of Mg²⁺.



Test for Zn²⁺

The supernatant solution at the end of the last test is analyzed for the presence of Zn^{2+} . Potassium hexacyanoferrate is added to a slightly acidic test solution. Formation of a white precipitate confirms the presence of Zn^{2+} .



Experimental Design

An unknown solution containing SIX cations belonging to Groups A, B, C, and D will be provided. The SIX cations must be identified using the procedures described below.

While testing the unknown sample, it is recommended to perform a test of known samples of ions within each group.

The procedures described below use a set of standard protocols that will be used during various stages of the analyses. These standard protocols and the preparation of the known samples for each analysis are described in this section.

1. Centrifuging the sample

Centrifuges will be used to separate a mixture into a solid precipitate and a liquid supernatant. The instructor will demonstrate the proper use of a centrifuge.

The centrifuge tubes are provided in the “CHEM 1C Additional Kit” provided by the stockroom. Only these tubes must be used inside the centrifuges. A tube of approximately the same mass in the opposite slot of the centrifuge must be used to balance the centrifuge. Be sure that centrifuge tubes are neither cracked nor chipped. The stress applied by the centrifuge can cause damaged tubes to shatter, resulting in chemicals and pieces of glass being scattered inside the centrifuge. A centrifuge without a top is dangerous; always close the top. Do not slow centrifuges down with your hands. They are spinning at a high rate of speed, and if there is any imperfection on the spinning surface, it can catch the flesh and do a great deal of damage in only an instant.

2. Separating a Precipitate from a Supernatant

After centrifuging, a supernatant can be separated from a precipitate by decanting. Carefully tip the centrifuge tube, and pour off the supernatant without disturbing the pellet of solid. The supernatant may be poured directly. Alternatively, the supernatant can be removed by carefully suctioning it up into a dropper.

3. Washing the precipitate

After separation from the supernatant, a precipitate is often washed to free it from reagents that might interfere at a later stage. Usually, the rinse is deionized water, but other liquids or solutions may be used. Add the indicated amount of the wash liquid and stir the contents of the test tube thoroughly. The pellet of

solid must be broken up and mixed well with the wash liquid. After thorough stirring, centrifuge the sample and decant the wash solution.

4. Adjusting and testing the pH

When directed to check the pH of a solution, stir the solution thoroughly with a clean glass stirring rod and then touch the tip of the rod to a piece of pH paper. Several such tests may be performed on each strip of paper. Never insert the test paper into the test tube, since the chemicals on the paper could contaminate the contents.

5. Heating

Due to the small quantity of material being heated, test tubes containing samples should NEVER be heated directly in a flame. A solution in test tube can reach its boiling point within a few seconds, and may be ejected violently from the test tube. All heating should be done using a water bath. Be careful that the tops of the test tubes are well above the water. The water may be boiling at times and could spatter into the test tubes, contaminating the contents. Labeling tape will fall off in a boiling water bath; therefore, it is best to label test tubes that are to be placed in a water bath with a sharpie marker.

6. Performing a flame test

When exposed to a flame, certain elements emit light of a characteristic color. Individual known sample solutions can be flame tested directly or the cation can be precipitated and a flame test performed on the precipitate.

In either case, a wire loop is used to introduce the sample into the flame. The loop of the flame test wire must first be thoroughly cleaned of any trace contamination. Begin by lighting a Bunsen Burner and adjusting the flame so that it burns hot; that is it appears blue, not yellow. Insert the wire loop into the hottest part of a Bunsen Burner flame; the tip of the inner blue cone. If the wire is contaminated, the flame will exhibit a color characteristic of the contaminant. Repeat the process of dipping the wire loop into DI water and then into the hot flame until no contamination is evident. Note that upon sufficient heating the test wire itself will turn the flame orange. After cleaning the wire, it can be used to test a solution or a precipitate.

To test a solution, use a clean dropper to remove one drop of the test solution and place this drop in the wire loop then insert it into the flame, observing the color that is emitted. For the known solutions, DO NOT insert the wire loop directly into the reagent bottle. Instead, place a small amount of the solution into a test tube for use. Each metal cation may not emit at the same burner temperature. Therefore, when performing flame tests best results are often obtained by slowly bringing the wire loop containing the sample into the flame

from the side. As the wire moves into the flame, it is subjected to a range of temperatures within the flame. This method of bringing the loop slowly into the flame is more important for the unknown samples, where concentrations of the emitting elements tend to be lower than in the known samples.

To flame test a precipitate, centrifuge and decant the supernatant. Wet the precipitate slightly with DI water. Dip a clean wire loop in the mixture containing the sample. If solid is present, attempt to get some of it to stick to the loop. Insert the wire loop into the flame, bringing the loop slowly into the flame from the side.

7. Known sample preparation: Group A

Combine 10 drops each of 0.1 M solutions of Ag^+ and Pb^{2+} in a centrifuge tube. Add three drops of 6 M HCl into the tube. Stir/shake the contents of the tube and wait for two minutes. Centrifuge the tube for two minutes. Discard the supernatant and test the precipitate for the Group A cations.

8. Known sample preparation: Group B

Obtain 10 drops each of 0.1 M solutions of Bi^{3+} , Fe^{3+} , Mn^{2+} , Al^{3+} , Cr^{3+} in a centrifuge tube. Add four drops of 6 M HCl followed by 6 M NH_3 until the pH of the solution is between 9 and 10. Centrifuge the tube for two minutes. Discard the supernatant. Wash the precipitate twice with 10 drops of deionized water. Add 10 drops of 6 M NaOH and two drops of 3% H_2O_2 and place the mixture in a boiling hot water bath for two minutes. Centrifuge the mixture for two minutes. Analyze the precipitate for the B1 cations and the supernatant for the B2 cations.

9. Known sample preparation: Group C

Obtain 10 drops each of 0.1 M solutions of Ba^{2+} , Ca^{2+} , Sr^{2+} in a centrifuge tube. Add four drops of 6 M HCl followed by 6 M NH_3 until the solution is neutral. Add an equal amount of NH_3 in excess. Centrifuge the mixture for two minutes. Analyze the supernatant solution for the Group C cations.

10. Known sample preparation: Group D

Obtain 10 drops each of 0.1 M solutions of Ni^{2+} , Cu^{2+} , Mg^{2+} , Zn^{2+} in a centrifuge tube. Add four drops of 6 M HCl followed by 6 M NH_3 until the solution is neutral. Add an equal amount of NH_3 in excess. Add five drops of 0.5 M ammonium oxalate. Stir the mixture and centrifuge for two minutes. Analyze the supernatant solution for the Group D cations.

Reagents and Supplies

0.1 M solutions of Ag^+ , Pb^{2+} , Bi^{3+} , Fe^{3+} , Mn^{2+} , Al^{3+} , Cr^{3+} , Ba^{2+} , Sr^{2+} , Ca^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} , Mg^{2+}

Acids: 6 M HCl, 6 M HNO₃, 6 M CH₃COOH

Bases: 6 M NH₃, 6 M NaOH

Other reagents: 1 M KI, 3% H₂O₂, NaBiO₃, SnCl₂, 0.1 M KSCN, 1 M NaHCO₃, 0.5 M (NH₄)₂C₂O₄, 3 M (NH₄)₂CO₃, 0.1 M K₂CrO₄, 0.1% dimethylglyoxime, 0.1 M K₄Fe(CN)₆, Na₂S₂O₃, 0.1 M Na₂HPO₄, 0.1% aluminon

(See posted Material Safety Data Sheets)

Procedure

PART 1: GROUP A (Ag^+ , Pb^{2+})

1. Obtain 10 drops of the unknown solution in a centrifuge tube.
2. Add three drops of 6 M HCl into the above centrifuge tube.
3. If a precipitate forms, then one of the group A cations is present in the unknown. In this case proceed to the next step. If no precipitate forms, then none of the group A cations are present and in this case proceed to Part 2.
4. Centrifuge the mixture containing the precipitate for two minutes. Decant the precipitate and save the supernatant for analysis in Part 2.
5. Wash the precipitate with 10 drops of deionized water two times.
6. Add 20 drops of deionized water to the precipitate and heat the mixture in a boiling hot water bath for three minutes.
7. Centrifuge the mixture again. If there is no precipitate, then confirm the presence of Pb^{2+} using the test described in Step 8. If there is a precipitate, then confirm the presence of Ag^+ using the test described in Step 9.
8. Test for Pb^{2+} : Add three drops of 1 M KI to the clear solution from Step 7. The formation of a yellow precipitate confirms the presence of Pb^{2+} .
9. Test for Ag^+ : Add 10 drops of 6 M NH_3 to the precipitate in Step 7. Stir the mixture. If Ag^+ is present, the precipitate will dissolve. Add 6 M HNO_3 to the same solution until a white precipitate of AgCl forms to confirm the presence of Ag^+ .

PART 2: GROUP B (Bi^{3+} , Fe^{3+} , Mn^{2+} , Al^{3+} , Cr^{3+})

1. Obtain the supernatant from Step 3 of Part 1. If additional test sample is needed, then repeat Steps 1-3 of Part 1.
2. Add 6 M NH_3 to the above sample until the pH is between 9 and 10. Centrifuge the mixture for two minutes. The precipitate will contain the Group B cations and the supernatant will contain the cations from Groups C & D. Save the supernatant for analysis in Part 3.
3. Wash the precipitate from Step 2 above twice with deionized water.
4. Add 10 drops of 6 M NaOH and two drops of 3% H_2O_2 to the precipitate, mix thoroughly and place in a boiling hot water bath for two minutes.
5. Centrifuge the above mixture for two minutes. The precipitate will contain the cations from Group B1 and the supernatant will contain the cations from Group B2. Test for the B1 cations according to procedures described in Steps 6-10. Test for the B2 cations according to procedures described in Steps 11-14.
6. Test for B1 cations: Add 2 mL of deionized water to the precipitate from Step 5. Mix thoroughly and heat for 10 minutes in a boiling hot water bath. This will remove any excess H_2O_2 from Step 4. Centrifuge the mixture and discard the supernatant.
7. Test for B1 cations: Add 10 drops of 6 M HCl to the precipitate. Mix and heat for 10 minutes in a boiling hot water bath. Centrifuge the mixture. The B1 cations will be found in the supernatant. Use the supernatant in the next three steps to test for each of the B1 cations. Discard the precipitate.
8. Test for Mn^{2+} : To five drops of the supernatant solution from Step 7, add several small amounts of NaBiO_3 until no further reaction is observed. Centrifuge the mixture and observe the color of the supernatant solution. A purple supernatant solution confirms the presence of Mn^{2+} .
9. Test for Bi^{3+} : To five drops of the supernatant solution from Step 7, add three drops of 6 M NaOH followed by a small amount of SnCl_2 . Formation of a black precipitate confirms the presence of Bi^{3+} .
10. Test for Fe^{3+} : To five drops of the supernatant from Step 7, add three drops of 0.1 M KSCN. Formation of a reddish brown complex confirms the presence of Fe^{3+} .
11. Test for B2 cations: Obtain the supernatant solution from Step 5 above and heat the solution for 10 minutes in a boiling hot water bath. Now, cool the solution in a cold bath. Add 6 M HCl until the solution is acidic and observe any color

changes. Add 1 M NaHCO_3 to neutralize the solution. Centrifuge the solution and divide the supernatant into two parts to be used in Steps 12 and 13.

12. Test for Al^{3+} : To two drops of the supernatant from Step 11, add two drops of 6 M HCl. Add two drops of aluminon followed by 6 M NH_3 until the solution is basic. Centrifuge the mixture for one minute. The appearance of a red precipitate confirms the presence of Al^{3+} .
13. Test for Cr^{3+} : To two drops of the supernatant from Step 11, add one drop of 3% H_2O_2 . Now add 6 M HCl until the solution is acidic. The appearance of a dark blue complex confirms the presence of Cr^{3+} . However, this complex decomposes quickly thereby causing the color to disappear.

PART 3: GROUP C (Ba^{2+} , Sr^{2+} , Ca^{2+})

1. Obtain the supernatant solution from Step 2 of Part 2. This contains the cations from Groups C & D. If additional test solution is required, then repeat Step 2 of part 2.
2. To the solution from Step 1 above, add five drops of 0.5 M ammonium oxalate and mix. The cations from Group C will precipitate and the cations from Group D will stay dissolved. Centrifuge this mixture for two minutes. Save the supernatant solution for analysis in Part 4.
3. Wash the precipitate with 1 mL of deionized water.
4. Transfer the precipitate into a crucible. Use concentrated HNO_3 to do the transfer. Cover the crucible partially with a lid. Heat the crucible over a low flame and evaporate the liquid.
5. Cool the crucible for five minutes and add six drops of concentrated HNO_3 . Cover the crucible partially with a lid. Heat the crucible over a low flame and evaporate the liquid.
6. Dissolve the precipitate in four drops of 6 M HCl . Transfer the solution to a clean test tube.
7. Test for Ba^{2+} : Perform the flame test with the solution from Step 6. Emission of yellow-green light confirms the presence of Ba^{2+} .
8. Test for Sr^{2+} : Perform the flame test with the solution from Step 6. Emission of a crimson light confirms the presence of Sr^{2+} .
9. Test for Ca^{2+} : Perform the flame test with the solution from Step 6. Emission of a brick-red light confirms the presence of Ca^{2+} .

PART 4: GROUP D (Ni^{2+} , Cu^{2+} , Zn^{2+} , Mg^{2+})

1. Obtain the supernatant solution from Step 2 of Part 3. This will contain the Group D cations.
2. Pour the supernatant liquid containing the Group D cations into a clean crucible. Place the lid on the crucible slightly ajar, and heat the solution to dryness over a low flame.
3. Cool the crucible for five minutes, and then add six drops of concentrated nitric acid, washing the inside of the crucible. Replace the lid and heat to dryness once again.
4. Cool the crucible for five minutes, repeat the addition of concentrated nitric acid and heat to dryness.
5. Cool the crucible for 5 minutes, and dissolve the residue by adding 5-10 drops of 6 M HCl. (It is alright if all of the solid does not dissolve.) Using a clean Pasteur pipet, transfer the solution to a clean test tube, label the test tube as "D".
6. Rinse the crucible with 5 drops of deionized water. Add this rinse to the test tube labeled "D".
7. Test for Ni^{2+} : Transfer one drop of the solution from test tube "D" to a clean test tube. Add 6 M NH_3 , with stirring, until the solution tests basic to litmus paper. Add one drop of 1% dimethylglyoxime. The appearance of a cherry red precipitate confirms the presence of Ni^{2+} .
8. Test for Cu^{2+} : Transfer one drop of the solution from test tube "D" to a clean test tube. Add 6 M NH_3 until the solution tests only weakly acidic. If the solution becomes basic, use 6 M acetic acid to make it slightly acidic. Add 3-4 drops of 0.1 M $\text{K}_4[\text{Fe}(\text{CN})_6]$, solution. The appearance of a maroon precipitate confirms the presence of Cu^{2+} .
9. Separation of $\text{Cu}^{2+}/\text{Ni}^{2+}$ from $\text{Mg}^{2+}/\text{Zn}^{2+}$: Add 6 M NH_3 to the remainder of the solution in test tube D until it tests slightly basic (pH 8-9). Then add 6 M acetic acid until the solution is weakly acidic (pH 4-5). Add about 0.2 g of solid sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, and heat for five minutes in a boiling water bath. If Cu^{2+} and/or Ni^{2+} are present, then a black precipitate should form. If a black precipitate does not form under these conditions, add one to two more drops of 6 M acetic acid and continue heating a few more minutes. (Formation of the black precipitate is pH sensitive.) Cool for 1 minute by swirling the test tube in cold tap water. Centrifuge and decant the supernatant into a clean test tube labeled D1. The precipitate may be discarded.

10. Test for Mg^{2+} : To the solution labeled D1 add 6 M NH_3 until the pH is about 8. Add 6-8 drops of saturated (about 0.1 M) Na_2HPO_4 solution. Stir and then cool the solution in ice water for several minutes. If a precipitate does not form, add 6 M HCl in drops to lower the pH slightly. Watch carefully for precipitate formation; stop adding the HCl upon formation of a precipitate. Centrifuge and discard the supernatant. The formation of a white precipitate indicates the possibility of Mg^{2+} .
11. Wash the precipitate with 10 drops of deionized water. Discard the wash. To the washed precipitate, add 6 drops of 6 M NaOH and stir thoroughly. Centrifuge and decant the supernatant into a clean test tube labeled D2.
12. Test for Zn^{2+} : To the solution in the test tube labeled D2 add 6 M acetic acid until the solution tests only weakly acidic (pH 4-5). If the solution becomes too acidic, use 6 M NH_3 to make it slightly acidic. Add 4 drops of 0.1 M $K_4[Fe(CN)_6]$. The formation of a white precipitate confirms the presence of Zn^{2+} .

Data Table

In each instance, clearly describe the observations and the appropriate inferences.

PART 1: GROUP A (Ag^+ , Pb^{2+})

Cation Analyzed	Observations from known	Observations from unknown	Inference about unknown
Pb^{2+}			
Ag^+			

PART 2: GROUP B (Bi^{3+} , Fe^{3+} , Mn^{2+} , Al^{3+} , Cr^{3+})

Cation Analyzed	Observations from known	Observations from unknown	Inference about unknown
Bi^{3+}			
Fe^{3+}			
Mn^{2+}			
Al^{3+}			
Cr^{3+}			

PART 3: GROUP C (Ba²⁺, Sr²⁺, Ca²⁺, Co²⁺)

Cation Analyzed	Observations from known	Observations from unknown	Inference about unknown
Ba²⁺			
Sr²⁺			
Ca²⁺			

PART 4: GROUP D (Ni²⁺, Cu²⁺, Zn²⁺, Mg²⁺)

Cation Analyzed	Observations from known	Observations from unknown	Inference about unknown
Ni²⁺			
Cu²⁺			
Zn²⁺			
Mg²⁺			

Results**STUDENT NAME:** _____**UNKNOWN NUMBER:** _____**The unknown provided consists of the following SIX cations:**

	Name	Formula
1		
2		
3		
4		
5		
6		