Laboratory

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Qualitative Analysis: Identification of Unknown Cations

Objectives

- Explore the solubility properties of salts
- Examine precipitation reactions
- Create a flowchart for determining ions present in unknown mixtures

Equipment and Materials

- 3 M nitric acid (HNO_3)
- 0.2 M potassium ferricyanide ($K_3Fe(CN)_6$)
- 0.3 M sodium phosphate (Na_3PO_4)
- 6 M sodium hydroxide (NaOH)
- 15 M ammonium hydroxide (NH_4OH)
- 0.1 M zinc nitrate ($Zn(NO_3)_2$)
- 0.1 M calcium nitrate ($Ca(NO_3)_2$)
- 0.1 M copper(II) nitrate ($Cu(NO_3)_2$)
- 0.1 M aluminum nitrate ($Al(NO_3)_3$)
- 0.1 M cobalt nitrate ($Co(NO_3)_2$)
- test tubes (large and small)
- Water bottle

Introduction

Qualitative analysis is a process used to find information regarding the composition of different species in a mixture. The analysis does not give us the amount of material present so it is not as rigorous as a quantitative analysis, but is often easier and faster to perform. In this lab you will be creating a way to identify the components in a mixture of aqueous metal ions using data gathered from several qualitative tests.

PART I

Data Collection

1. Set up a table in your lab notebook for observations similar to the one shown below. Be sure to make your grids large enough to write in your observations.
2. Choose one of the cations and place about 5 mL of its solution into a large test tube to serve as your stock solution for the tests on the following page. Be sure to label it so you know what is in there.

TEST	METAL				
	Al^{3+}	Ca^{2+}	Co^{2+}	Cu^{2+}	Zn^{2+}
$Fe(CN)_6^{3-}$					
PO_4^{3-}					
OH^-					
NH_3 (NH_4OH)					
H^+					

3. Perform the tests listed below (A–E) in order on your sample of metal ion. **Record your observations of each test in your table.**

   A. Place a fresh 10 drops of your metal cation solution into a new small test tube and add 4–6 drops of HNO_3 to acidify the solution. This is a necessary step before adding the potassium ferricyanide. Now add 7 drops of $K_3Fe(CN)_6$ solution to the test tube and record your observations. Dispose of this solution.

   B. Place a fresh 10 drops of your metal cation solution into a new small test tube. To this solution, add 7 drops of Na_3PO_4 and record your observations. Dispose of this solution.

   C. Place a fresh 10 drops of your metal cation solution into a new test tube. Add 8 drops of NaOH solution. If you observe a precipitate form in the test tube, mix the sample thoroughly and centrifuge your sample. After the sample is finished centrifuging, decant the liquid (*supernatant*) into a waste beaker and save the solid for the next step. If no precipitate is left from this step, you may skip tests D and E.

   D. Wash the solid from the previous step with ~1 mL of deionized water. Be sure to break up any solid with a glass stir rod to ensure a good wash. Centrifuge the tube again. Carefully decant the wash into your waste beaker, making sure you do not lose your solid. Repeat this wash procedure two more times. Once you have decanted your last wash, add 10 drops of NH_3 and mix for ~60 seconds. Record your results and dispose of this solution.

   E. Repeat the process in step C above to get a fresh precipitate from the hydroxide. Decant the supernatant into your waste beaker, and wash the precipitate two times as in the previous step. Once you have decanted your last wash, add 15 drops of HNO_3. Record your results and dispose of this solution.

Data Analysis

1. Use the observations in your data table to complete the following reactions for your lab report. If no reaction was observed, put NR. Assume that precipitates are simple cation:anion ratios and have a neutral charge. Include any reactions that occur in the data section of your report

A. Al^{3+}

A. $Al^{3+} + Fe(CN)_6^{3-} \rightarrow$

B. $Al^{3+} + PO_4^{3-} \rightarrow$

C. $Al^{3+} + OH^- \rightarrow$

D. $+ NH_3$ (if necessary) $\rightarrow$

E. $+ H^+$ (if necessary) $\rightarrow$

B. Ca^{2+}

A. $Ca^{2+} + Fe(CN)_6^{3-} \rightarrow$

B. $Ca^{2+} + PO_4^{3-} \rightarrow$

C. $Ca^{2+} + OH^- \rightarrow$

D. $+ NH_3$ (if necessary) $\rightarrow$

E. $+ H^+$ (if necessary) $\rightarrow$

C. Co^{2+}

A. $Co^{2+} + Fe(CN)_6^{3-} \rightarrow$

B. $Co^{2+} + PO_4^{3-} \rightarrow$

C. $Co^{2+} + OH^- \rightarrow$

D. $+ NH_3$ (if necessary) $\rightarrow$

E. $+ H^+$ (if necessary) $\rightarrow$

D. Cu^{2+}

A. $Cu^{2+} + Fe(CN)_6^{3-} \rightarrow$

B. $Cu^{2+} + PO_4^{3-} \rightarrow$

C. $Cu^{2+} + OH^- \rightarrow$

D. $+ NH_3$ (if necessary) $\rightarrow$

E. $+ H^+$ (if necessary) $\rightarrow$

E. Zn^{2+}

A. $Zn^{2+} + Fe(CN)_6^{3-} \rightarrow$

B. $Zn^{2+} + PO_4^{3-} \rightarrow$

C. $Zn^{2+} + OH^- \rightarrow$

D. $+ NH_3$ (if necessary) $\rightarrow$

E. $+ H^+$ (if necessary) $\rightarrow$

PART II

Interpretation

We now want to consider how we might use the data and information we've just collected to be able to sort out a solution that contains a mixture of ions that might include anywhere from one to all five of the cations used. Consider the following questions on how you might separate and verify a mixture of ions given to you.

1. Imagine that a classmate gives you a solution and tells you it contains only Ca^{2+} ions. Using the reaction data you collected, describe how you would prove or refute this claim. Write your answer in your lab notebook.
2. Now imagine that a classmate gives you a solution claiming it contains only Zn^{2+} and Cu^{2+} ions. Using your reaction data, describe how you would prove or refute that one, two, or possibly none of those ions are present. Write your answer in your lab notebook.
3. Next, imagine that a classmate gives you a solution claiming it contains only Al^{3+} and Co^{2+} ions. Using your reaction data, describe how you would prove or refute that one, two, or possibly none of those ions are present. Write your answer in your lab notebook.
4. For our final interpretation, imagine that you are given a solution that may contain any number of our five metal cations. Construct a flowchart on how you would separate the ions from each other and test for the presence of each one. **This flowchart needs to be completed by you and verified by your teaching assistant before leaving lab for the day.** Record your flowchart in your lab manual. *A sample set of data and flowchart for a different set of cations has been included to give you an idea of how you should proceed, and what a flowchart might look like. The flowchart should give no ambiguous results.*

PART III

Interpretation of Data from an Unknown Solution

1. You will be given a set of data by your teaching assistant for a solution that contains anywhere from two to four unknown metal ions from the group of five that you tested.
2. Use your flowchart to determine the contents of your unknown solution based on the data given.
3. In your Data Analysis section of your lab report, include all justification for the presence or absence of all the ions based on the data and flowchart.

SAMPLE OBSERVATION MATRIX

	Pb^{2+}	Fe^{3+}	Ni^{2+}	Ag^{+}	Ba^{2+}
CrO_4^{2-}	Bright yellow ppt	Light yellow ppt		Reddish ppt	Light yellow ppt
SCN^-		Dark red solution	Reddish solution		
OH^- (NH_4OH)		Dark red ppt	Green gel ppt	Gray ppt	Faint white ppt
DMG^-			Bright pink ppt		
Cl^- (HCl)	White ppt (dissolves when heated)	Darker yellow solution		White ppt	
SO_4^{2-} (H_2SO_4)	White ppt			Faint white ppt	White ppt

SAMPLE QUALITATIVE ANALYSIS FLOWCHART FOR SAMPLE OBSERVATION MATRIX ON PREVIOUS PAGE