EXPERIMENT 15C

Qualitative Analysis Scheme of Main Group and Transition Metal Cations without Hazardous Waste*

The following experiment is intended to continue the introduction of qualitative analysis through the identification of various main group and transition metal cations. Recall that the analysis of a substance can be extremely difficult given the fact that there are thousands of possibilities for reactions with some similar results. For example, you may have seen from the Group I qualitative analysis that Ag^{+}, Pb^{+2}, and Hg_2^{+2} all produce a white precipitate when Cl^{-} is added.

In this experiment, the ions initially chosen were limited to aluminum, the alkaline earth metals, and certain first-row transition metals with the goal of minimizing the amount of hazardous waste generated. Analysis of both a known and unknown solution will test for Al^{+3}, Ba^{+2}, Ca^{+2}, Co^{+2}, Cu^{+2}, Fe^{+3}, Mg^{+2}, Mn^{+2}, and Ni^{+2}. The ions are roughly divided into three groups. The group A ions have very insoluble oxides or hydroxides. The group B ions are poorly coordinated by NH_3 and have very insoluble carbonates; magnesium is included in group B even though it is precipitated separately. Group C metal ions are strongly coordinated by NH_3. This prevents their precipitation in the previous steps.

Note that almost all of the solutions can safely go down the sink in moderate amounts. The barium sulfate should be collected, but its storage and disposal should not pose a serious problem in the small amounts used in the tests. The organic compounds used to test for the transition metal ions should be handled with care as they are irritants and suspected carcinogens. Once in solution, they are used in small amounts that should not pose a serious hazard.

PROCEDURE:

Separation of Ion Groups: Unknown and Known (with each group)

1. Place 1 mL of your test solution in a medium test tube.

2. Add l2 drops of 6 M HNO_3, 3 mL of 6 M NH_3(aq), and stir. Centrifuge and decant. The solid may contain $Al(OH)_3$, $Fe(OH)_3$, and $Mn(OH)_2$. *Save the solid for Group A analysis*. Use the solution in the next step.

3. To the solution from step 2, add 2 mL, of 1 M $(NH_4)_2CO_3$ solution and stir. Centrifuge and decant. The solid may contain $BaCO_3$ and $CaCO_3$. *Save the solid for Group B analysis.* Use the solution in the next step.

4. To the solution from step 3, add 1 mL of 1 M Na_2HPO_4 solution. A white precipitate of $MgHPO_4$ indicates the presence of magnesium ion. Centrifuge and decant. *Save the solution for Group C analysis*.

$$Mg^{+2}(aq) + HPO_4^{-2}(aq) \rightarrow MgHPO_4(s) \quad (1)$$

Analysis of the Groups of Ions: Group A (Al^{3+}, Fe^{3+}, Mn^{2+})

1. Add 1 mL of 6 M NaOH to solid A from step 2 and stir vigorously with a glass stirring rod. Centrifuge and decant. In strong base, $Al(OH)_3$ solid will separate into the solution, forming the complex ion $Al(OH)_4^-$.

2. To the solution, add 2 M acetic acid drop wise until **slightly** acidic. A white precipitate indicates aluminum ion.

$$Al(OH)_4^-(aq) + H^+(aq) \rightarrow Al(OH)_3(s) + H_2O(l) \quad (2)$$

3. To the solid from step 1, add 2 mL of H_2O. Then add 6 M HNO_3 drop wise until acidic to dissolve the solid from step 1 in order to isolate Fe^{+3} and Mn^{+2} ions. Divide the solution into two test tubes.

4. To one test tube, add 2 -3 drops of 0.5 M KSCN. A dark-red solution indicates iron(III) ion.

$$Fe^{+3}(aq) + SCN^-(aq) \rightarrow FeSCN^{+2}(aq) \quad (3)$$

5. To the other test tube, add a spatula tip of $NaBiO_3 \cdot 5H_2O$. A purple solution indicates manganese(II) ion has oxidized to MnO_4^-. (The color of the solution will fade on standing as it reacts in the air.)

$$2Mn^{+2}(aq) + 14H^+(aq) + 5BiO_3^-(aq) \rightarrow \underset{\text{purple}}{2MnO_4^-(aq)} + 5Bi^{+3}(aq) + 7H_2O \quad (4)$$

Group B (Ba^{2+}, Ca^{2+})

<u>NOTE</u>: *Magnesium ion is technically part of Group B. However, the test for magnesium ion has already been performed in step 4 of the ion group separation.*

1. Add 1 mL of water to solid B from step 3 of the ion group separation, and then slowly add 6 M HCl dropwise. Stir until the solid has dissolved.

2. Add 1 mL of l M Na_2SO_4 and stir. A white precipitate indicates barium ion. Centrifuge and decant.

$$Ba^{+2}(aq) + SO_4^{-2}(aq) \rightarrow BaSO_4(s) \quad (5)$$

3. To the solution, add 3 drops of 6 M NH_3(aq) and 1 mL of 0.3 M $(NH_4)_2C_2O_4$ solution. Stir and let stand for 1 min. The formation of a white precipitate indicates calcium ion.

$$Ca^{+2}(aq) + C_2O_4^{-2}(aq) \rightarrow CaC_2O_4(s) \qquad (6)$$

Group C (Co^{2+}, Cu^{2+}, Ni^{2+})

1. Pour the solution for Group C analysis containing $Co(NH_3)_6^{+2}$, $Cu(NH_3)_4^{+2}$, and $Ni(NH_3)_6^{+2}$ from step 4 of the ion group separation into casserole and heat gently to dryness. If a dark residue remains, continue heating until a pale solid remains.

2. Dissolve the solid from step 1 in 2 mL of 1 M H_3PO_4. Then add 1 mL of 1 M Na_2HPO_4 and divide it equally into three test tubes.

3. To the first test tube add a spatula tip of KI and stir. (Alternatively, add 2-3 drops of 0.5 M KI if available.) A brown solution with a pale tan precipitate indicates copper(II) ion.

4. To the second test tube add Na_2HPO_4 until basic and then add a few drops of 1% dimethylglyoxime solution (DMG^-). A red/pink precipitate indicates nickel ion.

$$Ni(NH_3)_6^{+2}(aq) + 3DMG^-(aq) \rightarrow Ni(DMG)_3^-(aq) + 6NH_3(g) \qquad (7)$$

5. To the third test tube, add 1 mL of 1% 1-nitroso-2-naphthol in 95% ethanol solution and stir. A red-brown precipitate indicates cobalt(II) ion.

NAME _____ SECTION_____

EXPERIMENT 15C – General Qualitative Analysis
REPORT SHEET

UNKNOWN NUMBER: _____ANALYSIS _____

YOUR FLOW CHART: **(Make the chart complete by showing all steps, even washings, all reagents at proper concentrations and their amounts, color of precipitates, and any other information that was interesting and/or important.)**

DISCUSSION QUESTIONS:

1. The precipitates of iron (III) ion and manganese (II) ion are hydroxides. What is the purpose of adding 6 M HNO_3(aq) in step 3 of the Group A analysis?

2. In step 2 of Group B, Na_2SO_4 is added. A white precipitate indicates the presence of barium ion. Suggest a reason why a calcium precipitate does not form along with the barium precipitate.

3. Describe any modifications (if any) that you discovered that worked for your analysis. How would you improve this experiment?