**Separation and Qualitative Determination of Cations and Anions**

**Purpose:** The purpose of this lab is to identify the cations and anions components in the unknown solution. This can be done by using the method called selective precipitation of ions. This uses the solubility rule to dissolve and precipitate the desired ions. The significance of this lab is that forensic scientist can use these skills to identify the components of the substance found in the corpse.

**Hypothesis:** The hypothesis is that the component ions of the solution can be identified. This is because each ion has different solubility conditions. Thus, using their difference in their solubility, the ions can be separated through the method of selective precipitation. After the separation, the ions can be confirmed through other means such as reacting with other substances to see the color change and etc.

**Materials:**

<table>
<thead>
<tr>
<th>Materials</th>
<th>Quantities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>25 mL</td>
</tr>
<tr>
<td>0.2 M Silver nitrate (AgNO₃)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Copper(II) nitrate (Cu(NO₃)₂)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Zinc nitrate (Zn(NO₃)₂)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Iron(III) nitrate (Fe(NO₃)₃)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Manganese(II) nitrate (Mn(NO₃)₂)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Aluminum nitrate (Al(NO₃)₃)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Potassium iodide (KI)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Sodium bromide (NaBr)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Sodium carbonate (Na₂CO₃)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Sodium chloride (NaCl)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Sodium nitrate (NaNO₃)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.2 M Sodium sulfate (Na₂SO₄)</td>
<td>5 mL</td>
</tr>
<tr>
<td>6 M Sodium hydroxide (NaOH)</td>
<td>8 mL</td>
</tr>
<tr>
<td>6 M Sulfuric acid (H₂SO₄)</td>
<td>10 mL</td>
</tr>
<tr>
<td>6 M Nitric acid solution (HNO₃)</td>
<td>10 mL</td>
</tr>
<tr>
<td>6 M Acetic acid solution (CH₃COOH)</td>
<td>10 mL</td>
</tr>
<tr>
<td>6 M Hydrochloric acid (HCl)</td>
<td>10 mL</td>
</tr>
<tr>
<td>6 M Ammonia solution (NH₃)</td>
<td>10 mL</td>
</tr>
<tr>
<td>0.1 M Barium chloride (BaCl₂)</td>
<td>10 mL</td>
</tr>
<tr>
<td>0.1 M Silver nitrate (AgNO₃)</td>
<td>10 mL</td>
</tr>
<tr>
<td>0.1 M Potassium ferrocyanide (K₃Fe(CN)₆)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.1 M Potassium permanganate (KMnO₄)</td>
<td>5 mL</td>
</tr>
<tr>
<td>0.1 M Potassium thiocyanate (KSCN)</td>
<td>5 mL</td>
</tr>
<tr>
<td>Aluminum granules (Al)</td>
<td>About 0.05 g</td>
</tr>
<tr>
<td>Saturated barium hydroxide (Ba(OH)₂)</td>
<td>10 mL</td>
</tr>
<tr>
<td>0.1% Aluminon</td>
<td>5 mL</td>
</tr>
<tr>
<td>3% Hydrogen peroxide (H₂O₂)</td>
<td>5 mL</td>
</tr>
<tr>
<td>Mineral oil</td>
<td>5 mL</td>
</tr>
</tbody>
</table>
Sodium bismuthate (NaBiO₃) 1 g
0.6 M Iron (III) nitrate (Fe(NO₃)₃ in 0.1 M HNO₃ 5 mL
600-mL beaker 1 beaker
250-mL beaker 1 beaker
Boiling tube 20 tubes
Test tube rack 2 racks
Test tube 12 test tubes
Test tube stoppers 2 stoppers
Centrifuge 1 centrifuge
Litmus paper 5 of blue and 5 of pink litmus paper
Spatula 2 spatula
Centrifuge tube 5 centrifuge tubes
Hot plate with magnetic stirrer 1 hot plate with magnetic stirrer

Procedures:

Part 1. Qualitative Analysis of Cations

Step 1. Separation of Silver from Iron, Copper, Aluminum, Manganese, and Zinc Ions

1.) Pour the solution to be analyzed in a centrifuge pellet
2.) Add 8 drops of 6 M HCl to the solution to be analyzed
3.) Stir the solution
4.) A white precipitate indicates that the Ag⁺ ion is present
5.) Centrifuge the solution
6.) Add one more drop of 6 M HCl to make sure the precipitation is complete
7.) Centrifuge the solution again
8.) Pour off the liquid on the top and save the liquid in a second centrifuge pellet for step #3
9.) Wash the precipitate by adding 1 mL distilled water
10.)Centrifuge and discard the wash water
11.)Save the precipitate for step #2

** Since no precipitate formed, the separation of manganese, iron, and copper from aluminum and zinc was completed next **

Step 3. Separation of Manganese, Iron, and Copper from Aluminum and Zinc

1.) To the solution saved from step #1.8., add 10 drops of 3% hydrogen peroxide, H₂O₂
2.) Stir the solution
3.) Add 6 M sodium hydroxide, NaOH, until the solution is basic
4.) Add 3 more drops of NaOH
5.) Stir and place the solution in a hot water bath for 3 minutes
6.) The formation of a precipitate indicates the presence of either copper, iron, manganese, or a combination of all three
7.) Centrifuge the solution
8.) Separate the clear solution from the solid
9.) Save the clear solution for step #9
10.) Wash the precipitate with a mixture of 10 drops of 6 M NaOH and 10 drops of water
11.) Centrifuge and discard the wash water
12.) Save the precipitate for step #4

**Step 4. Separate Manganese Ions from Iron and Copper Ions**

1.) Add 5 drops of water to the precipitate from step #3.12.
2.) Add 6 M H$_2$SO$_4$ dropwise until the solution is acidic when tested with litmus paper
3.) Centrifuge and separate the MnO$_2$ precipitate
4.) Save the solution for step #5
5.) Wash the precipitate with water
6.) Centrifuge the solution
7.) Discard the wash water
8.) Save the precipitate for step #7

**Step 5. Separation of Iron from Copper; Confirmation of Copper**

1.) To the solution from step #4.4., add 6 M aqueous NH$_3$ until the solution is basic to litmus
2.) Add an extra mL of NH$_3$ to the solution
3.) Centrifuge and separate the supernatant liquid from the precipitate
4.) Save the precipitate for step #6
5.) The presence of the blue Cu(NH$_3$)$_4^{2+}$ ion is the confirmatory test for copper
6.) Dispose the copper solution

**The addition of NH$_3$ did not produce any precipitate, therefore, identification of manganese ions will be completed next**

**Step 7. Manganese Ions**

1.) To the precipitate from step #4.8., add 1 mL of water and 1 mL of 6 M H$_2$SO$_4$
2.) Add 1 mL of 3% H$_2$O$_2$
3.) Heat the solution in a boiling water bath and stir
4.) Wait for the precipitate to dissolve

**Step 8. Confirmation of Manganese**

1.) To the solution from step #7.4., add 1 mL of 6 M HNO$_3$
2.) Add in a spatula of solid sodium bismuthate, NaBiO$_3$
3.) Add more NaBiO$_3$ until the excess remains
4.) Stir and centrifuge the solution
5.) The purple color of the MnO$_4^-$ ion in the solution confirms the presence of manganese
6.) Dispose the manganese
**Step 9. Separation of Zinc and Aluminum**

1.) To the colorless solution from step #9., add about 10 drops of 6 M HNO₃
2.) Test to see if the solution is acidic
3.) Continue to add HNO₃ until the solution is acidic
4.) Add 6 M NH₃ dropwise until the solution is basic to litmus
5.) Add an additional 3 drops of the 6 M NH₃
6.) Stir the solution
7.) Centrifuge the solution and carefully separate the supernatant liquid from the precipitate

**Addition of NH₃ did not result in precipitate forming, therefore, confirmation of zinc will be the next step**

**Step 11. Confirmation of Zinc**

1.) Make the solution from step #9.7 slightly acidic by adding 6 M HCL drop wise
2.) Add 3 drops of 0.1 M K₄[Fe(CN)₆] and stir
3.) Centrifuge to see the confirmatory precipitate of K₂Zn₃[Fe(CN)₆]₂, which is white to light green or blue green in color
4.) Dispose the zinc precipitate and solution

**Part 2. Qualitative Analysis of Anions**

**Step 1. Separation of the Halides (Cl⁻, Br⁻, I⁻); Confirmation of Chloride**

1.) Place 10 drops of “Serena” test solution into a test tube
2.) Dip a stirring rod into the solution
3.) Test whether the solution is acidic using the litmus paper
4.) If the solution is not acidic, add 6 M CH₃COOH drop wise until the solution is acidic
5.) Add 10 drops of AgNO₃
6.) Transfer 6 drops into a centrifugal tube
7.) Centrifuge the solution
8.) Use the pipette to remove the supernatant liquid from the centrifugal tube
9.) Add 0.5 mL of distilled water into the centrifugal tube
10.) Centrifuge the solution
11.) Dump the supernatant distilled water
12.) Add 0.5 mL of 6 M NH₃
13.) Stir the solution
14.) Centrifuge the solution
15.) Transfer the supernatant liquid into another test tube
16.) Discard the precipitate (AgBr and AgI) if any
17.) Add 1 mL of 6 M nitric acid to the supernatant liquid in the test tube
18.) Test whether the solution is acidic using the litmus paper
19.) If not acidic, add HNO₃ drop wise until the solution becomes acidic
20.) If white precipitate forms, the presence of chloride is confirmed
**The addition of NH$_3$ did not produce and precipitates, thus, the experiment will proceed to the confirmation of carbonate**

**Step 3. Confirmation of Carbonate**

1. Shake the Ba(OH)$_2$ solution to make the solution becomes saturated
2. Wait for about 10 minutes
3. Place 1 mL of clear Ba(OH)$_2$ solution into a test tube
4. Pour 2 mL of clear Ba(OH)$_2$ solution into another test tube
5. Add 0.5 mL of 6 M HNO$_3$ into the test tube with “Serena” solution
6. Place the tube in a hot water bath
7. Use a dry Beral-type pipet to draw the gas released from the “Serena” solution
8. Dip the pipette in the test tube with Ba(OH)$_2$ solution
9. Squeeze the gas out of the pipette into the solution
10. Repeat step 7 to 9 for five times
11. The formation of white precipitate forming in the Ba(OH)$_2$ solution confirms the presence of carbonate

**Step 4. Confirmation of Sulfate**

1. Place 0.5 mL of the “Serena” test solution in a test tube
2. Use the litmus paper and check whether the solution is acidic or not
3. If not, add 6 M HNO$_3$, drop wise until the solution is acidic
4. Add 0.5 mL of 0.1 M BaCl$_2$ solution
5. The formation of white precipitate confirms the presence of sulfate

**All three unknown anions have been found and therefore confirmation of nitrate was no longer needed**

**Results:**

**Cations Observation:**

1. **Separation of the Silver from Iron, Copper, Aluminum, Manganese, and Zinc Ions**

   The cation solution was clear prior to the addition of HCl. After the addition of 8 drops of HCl, the solution remained clear, with no precipitate.

3. **Separation of Manganese, Iron, and Copper from Aluminum and Zinc**

   After the addition, of 10 drops of 3% hydrogen peroxide to the clear solution from step 1, a rapid change involving the formation of a dark brown precipitate and bubbles formation took place. The vigorous reaction caused the bubbles to spill over the top of the centrifuge pellets, causing deep brown stains on the surrounding tables. The addition of 6 M NaOH to the now-brown solution caused the solution to turn basic. No change overcame the solution after it was placed into the water bath for 3 minutes; the
solution was still brownish. The solution was separated into two layers after it was centrifuged: the brown-blackish floating substances all settled to the bottom while the solution went back to its original clear state.

4.) Separate Manganese Ions from Iron and Copper Ions

The addition of 6 M $\text{H}_2\text{SO}_4$ and distilled water to the precipitate from step 3 made the precipitate solution acidic, but no change was done to the precipitate itself.

5.) Separation of Iron from Copper: Confirmation of Copper

The addition of 6 M $\text{NH}_3$ to the supernatant solution from step 4 made no change to the solution; no precipitate was formed, and the color of the solution remained clear.

7.) Manganese Ions

No change happened to the precipitate from step 4 after the addition of 1 mL distilled water, 1 mL 6 M $\text{H}_2\text{SO}_4$, and 1 mL 3% $\text{H}_2\text{O}_2$. The precipitate gradually began to dissolve after the solution was placed into the boiling water bath. The resulting solution was clear and full of gas bubbles.

8.) Confirmation of Manganese

The solution from step 7 experienced no changes after the addition of 1 mL 6 M $\text{HNO}_3$. The addition of solid sodium bismuthate, a yellow powdery substance, caused the solution to undergo a sudden reaction that resulted in the rapid formation of white-yellowish bubbles, similar to the reaction between the cation solution and $\text{H}_2\text{O}_2$ in step 3. Excess solid sodium bismuthate settled to form a collective precipitate at the bottom of the pellet. After the reaction between the solution and sodium bismuthate subdued, the solution had a yellow-greenish color. There was no trace of the color purple in the solution even after it was centrifuged.

9.) Separation of Zinc and Aluminum

The clear solution from step 3 turned acidic after about 10 drops of 6 M $\text{HNO}_3$ were added. The solution then turned basic after a few drops of $\text{NH}_3$ were added. Other than the pH changes, no apparent changes overcame the solution, and no precipitate formed.

11.) Confirmation of Zinc

The solution became acidic again after the addition of 6 M $\text{HCl}$. It then turned a light green color, with the formation of a blue-whitish precipitate after 0.1 M $\text{K}_4[\text{Fe(CN)}_6]$ was added. The precipitate became easier to see after the solution is centrifuged, but the solution continued to have its original light-green color.

Anions Observation:
1.) Separation of Halides (Cl\(^-\), Br\(^-\), I\(^-\)); Confirmation of Chloride

The original unknown solution “Serena” has a slightly pink color. The solution becomes acidic after two drops of CH\(_3\)COOH was added. After the addition of 10 drops of AgNO\(_3\), the solution turns milky white. Once centrifuged, the supernatant solution was clear while leaving a small amount of white powder-like precipitate at the bottom. The precipitate dissolves completely in 6 M NH\(_3\). No precipitate can be found after centrifuging the solution. The addition of 6 M HNO\(_3\) caused the solution to become slightly warm, smoke was produced, and the solution turns milky. No precipitate forming after the addition of 6 M NH\(_3\) shows that there were no AgBr or AgI in the solution.

3.) Confirmation of Carbonate

The solution of Ba(OH\(_2\)) was clear with white precipitates at the top and bottom of the solution. Once shook, the solution turns milky white color. After some time to let the precipitate settles, the precipitate was once again at the top and bottom of the boiling tube. However, the solution was not as clear as before. The solution has some small white precipitate being suspended in it, causing the solution to look like some sort of hair gel. No changes occurred except bubbles forming when the “Serena” solution was put in hot water bath. Drawing the gas from the “Serena” solution caused the inside of the bulb of the Beral-type pipette to become slightly foggy. The change in the color of the Ba(OH\(_2\)) was very slow, but after about five times, the Ba(OH\(_2\)) solution turns white.

4.) Confirmation of Sulfate

It took one drop of 6 M HNO\(_3\) to turn the solution acidic. Adding 0.1 M BaCl\(_2\) turns the solution white.

<table>
<thead>
<tr>
<th>Step Number</th>
<th>Unknown Solution “y +”</th>
<th>Conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.4</td>
<td>No precipitates formed</td>
<td>The solution does not contain Ag(^+)</td>
</tr>
<tr>
<td>3.1</td>
<td>Dark brown precipitate and bubbles form</td>
<td>Either copper, iron, manganese, or the combination of all three ions exist in the solution</td>
</tr>
<tr>
<td>3.7</td>
<td>The solution was separated into two layers: the brown-blacking precipitate and the clear solution</td>
<td>Either copper, iron, manganese, or the combination of all three ions exist in the solution</td>
</tr>
<tr>
<td>5.2</td>
<td>The color of the solution remains clear</td>
<td>Cu(^{2+}) is not present in the solution</td>
</tr>
<tr>
<td>8.2</td>
<td>Rapid formation of white-yellowish bubbles occurs. The solution turns yellow-greenish color</td>
<td>Some reactions are occurring</td>
</tr>
<tr>
<td>9.5</td>
<td>No precipitate forms</td>
<td>The solution does not contain aluminum ions</td>
</tr>
<tr>
<td>11.2</td>
<td>The solution turns light green, blue-whitish precipitate forms</td>
<td>Precipitate indicates the presence of Zinc ions</td>
</tr>
</tbody>
</table>
### Part 2. Qualitative Analysis of Anions

<table>
<thead>
<tr>
<th>Step Number</th>
<th>Unknown Solution “Serena –“</th>
<th>Results</th>
<th>Conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5</td>
<td>Precipitate forms</td>
<td>Either I, Br, Cl, or all is present in the solution</td>
<td></td>
</tr>
<tr>
<td>1.12</td>
<td>The precipitate dissolves completely</td>
<td>Br and I were not present in the solution</td>
<td></td>
</tr>
<tr>
<td>1.20</td>
<td>White precipitate forms</td>
<td>Cl⁻ ions are present in the solution</td>
<td></td>
</tr>
<tr>
<td>3.6</td>
<td>Bubbles form in the solution</td>
<td>Gas is being produced</td>
<td></td>
</tr>
<tr>
<td>3.11</td>
<td>The Ba(OH)₂ solution turns from clear to white</td>
<td>Carbonate exists in the solution</td>
<td></td>
</tr>
<tr>
<td>4.4</td>
<td>The solution turns white</td>
<td>Sulfate is present in the solution</td>
<td></td>
</tr>
</tbody>
</table>

### Composition Comparison Table

<table>
<thead>
<tr>
<th></th>
<th>Experimented Composition</th>
<th>Actual Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>y + solution</td>
<td>Mn(NO₃)₂</td>
<td>Zn(NO₃)₂</td>
</tr>
<tr>
<td>Serena - solution</td>
<td>NaCl</td>
<td>Na₂CO₃</td>
</tr>
</tbody>
</table>

**Analysis:** The hypothesis could somewhat be verified as true. All the three anion components of the “Serena” solution have been identified. As for the “y” solution, the zinc component has been identified. Manganese was also identified due to reactions occurring but does not show the purple color. The precipitating reagent for Ag⁺ includes Br⁻, I⁻, as well as Cl⁻. NaCl would work well because the NaCl is a strong electrolyte and will ionize into Na⁺ and Cl⁻. Also, Na⁺ would be a spectator ion because it is always soluble and, therefore, does not affect the reaction between Ag⁺ and Cl⁻.

\[
\text{Cl}^- (aq) + \text{Ag}^+(aq) \rightarrow \text{AgCl(s)}
\]

\[
\text{AgCl(s)} + 2\text{NH}_3(aq) \rightarrow \text{Ag(NH}_3)_2^+(aq) + \text{Cl}^- (aq)
\]

\[
\text{Ag(NH}_3)_2^+(aq) + \text{Cl}^- (aq) + 2\text{H}^+(aq) \rightarrow \text{AgCl(s)} + 2\text{NH}_4^+(aq)
\]

\[
\text{Fe}^{3+}(aq) + 3\text{OH}^-(aq) \rightarrow \text{Fe(OH)}_3(s)
\]

\[
\text{Cu}^{2+}(aq) + 4\text{NH}_3(aq) \rightarrow \text{Cu(NH}_3)_2^2+(aq)
\]

Oxidation: \(\text{H}_2\text{O}_2(aq) \rightarrow 2\text{H}^+(aq) + \text{O}_2(g) + 2\text{e}^-\)

Reduction: \(\text{H}_2\text{O}_2(aq) + 2\text{H}^+(aq) + 2\text{e}^- \rightarrow 2\text{H}_2\text{O}(l)\)

Oxidation - the oxidation number of manganese increases from 2+ to 4+.
Mn(OH)_2(s) + H_2O_2(aq) → MnO_2(s) + H_2O(l)

Reduction – the oxidation number of manganese decreases from 4+ to 2+

MnO_2(s) + H_2O_2(aq) + 2H^+(aq) → Mn^{2+}(aq) + O_2(g) + 2H_2O(l)

Oxidation – the oxidation number of manganese increases from 2+ to 7+

2Mn^{2+}(aq) + 14H^+(aq) + 5BiO_3^-(aq) → 2MnO_4^-(aq) + 5Bi^{3+}(aq) + 7H_2O(l)

It was necessary to test for iodide by oxidation with Fe^{3+} first because the MnO_4^- would show a positive result if at least one of the anion, Br^- or I^-, is present in the solution. Therefore, it would be unknown whether the solution contains only I^-, only Br^-, or both ions. Half-reaction

Oxidation half – reaction: NO_3^-(aq) + 6H_2O(l) + 8e^- → NH_3(aq) + 9OH^-(aq)

Reduction half – reaction: Al(s) + 4OH^-(aq) → Al(OH)_4^-(aq) + 3e^-}

Care must be taken to keep the moist litmus from coming in contact with the cotton or the solution because the solution is basic. If the litmus paper were to come into contact with the basic solution, its color would change from red to blue whether or not NO_3^- is present in the solution. Therefore, the litmus paper must be kept away from the solution. This allows the litmus paper to change color only if NH_3 gas is from produced from the nitrate ions and nitrate ion is present in the solution. BaCO_3 did not precipitate in step 4, but it does in step 3 because of the surrounding condition. In step 4, the solution that the reaction occurred in was acidic from the addition of 6 M HNO_3. On the other hand, the solution in step 3 was basic because the Ba(OH)_2 is a strong base. BaCO_3 precipitate in step 3 because Ba^{2+} reacts with CO_3 in a strongly basic solution.

**Conclusion:** The hypothesis can be verified. Cations and anions have been identified through the process of selective precipitation. There are some errors, however, during the course of the experiment causing some problems in the identification of cations. An error could be that when sodium bismuthate was added to the solution of manganese ion, the reaction was rapid and causes some solution to spill out of the centrifuge pellets. Thus, this might have caused a decrease in the amount of manganese ions. Consequently, the decrease in the amount of manganese ions could have caused the purple color of the manganese ions to be light and become overshadowed by the color of the bismuthate or bismuth ions. Another error could be that there were some problem with the Fe(NO_3)_3 solution. Perhaps the solution was too diluted because some mass of Fe(NO_3)_3 was lost during the transfer from the wash glass to the graduated cylinder. This may have caused the solution to be too diluted to give any significant result when tested. There are ways to solve the errors mentioned. The first error could be solved by reacting the solution and sodium bismuthate in a test tube, preventing the reaction from spilling. The second error could be solved by using a better transfer method such as washing the remains of Fe(NO_3)_3 solids from the wash glass into the graduated cylinder. After that, the solution should be tested to see whether the Fe(NO_3)_3 solution made will give a positive result in the Fe test.