**General Chemistry II Laboratory**

**Experiment #3 Inorganic Qualitative Analysis**

**Separation and Characterization of Group B Cations**

**Introduction:** In this experiment the cations of the Group B metals Al, Cr and Sn will be characterized. The cations are in the form Al(OH)$_4$$^{-1}$(aq), CrO$_4$$^{-2}$(aq), and Sn(OH)$_6$$^{-2}$(aq) in the solution saved from the previous Group A Cation Analysis experiment.

The unknown solution is made acidic with HCl, taking care to note the appearance of blue color that rapidly fades. This may happen if chromate is present and all the hydrogen peroxide has not been destroyed. (See test for chromium). The reactions that occur are shown below.

\[
\text{Al(OH)}_4^{-1}(aq) + 4H^+^{1}(aq) \rightarrow \text{Al}^{3+}(aq) + 4H_2O(l)
\]
\[
\text{Sn(OH)}_6^{2-}(aq) + 6H^+^{1}(aq) + 6Cl^{-1}(aq) \rightarrow \text{SnCl}_6^{2-}(aq) + 6H_2O(l)
\]
\[
2\text{CrO}_4^{2-}(aq) \text{(yellow)} + 2H^+^{1}(aq) \rightarrow \text{Cr}_2\text{O}_7^{2-}(aq) \text{(orange)} + H_2O(l)
\]

This solution is next treated with bicarbonate to neutralize the acid. Tin(IV) ion precipitates as SnO$_2$, while dichromate and aluminum ions are unaffected.

\[
\text{SnCl}_6^{2-}(aq) + 2H_2O(l) + 4NH_3(aq) \rightarrow \text{SnO}_2(s) + 4NH_4^{+1}(aq) + 6Cl^{-1}(aq)
\]

**Tin (IV) Ion:** Solid SnO$_2$ is treated with HCl to dissolve the precipitate. Aluminum metal is then added to convert the solid tin(II) oxide to SnCl$_4^{2-}$.

\[
\text{SnO}_2(s) + 4H^+^{1}(aq) + 6Cl^{-1}(aq) \rightarrow \text{SnCl}_6^{2-}(aq) + 2H_2O(l)
\]
\[
3\text{SnCl}_6^{2-}(aq) + 2\text{Al}(s) \rightarrow 3\text{SnCl}_4^{2-}(aq) + 2\text{Al}^{3+}(aq) + 6\text{Cl}^{-1}(aq)
\]

When aluminum metal is added to an acidic solution, bubbling will occur due to the formation of hydrogen gas.

\[
2\text{Al}(s) + 6H^+^{1}(aq) \rightarrow 2\text{Al}^{3+}(aq) + 3H_2(g)
\]

Mercury (II) is then added which oxidizes Sn$^{2+}$ back to Sn$^{4+}$. Hg$^{2+}$ is reduced to Hg$_2^{2+}$, mercury(I), which precipitates as the white-colored Hg$_2$Cl$_2$, confirming Sn$^{2+}$.

\[
\text{SnCl}_4^{2-}(aq) + 2\text{Hg}^{2+}(aq) + 4\text{Cl}^{-1}(aq) \rightarrow \text{Hg}_2\text{Cl}_2(s) \text{(white)} + \text{SnCl}_6^{2-}(aq)
\]

**Aluminum (III) Ion:** The remaining supernatant liquid is divided into two parts to test for aluminum and chromium. The Al$^{3+}$ ion is precipitated as Al(OH)$_3$ in the presence of the red dye aluminon. The red dye is adsorbed on the surface of the aluminum hydroxide precipitate. A red precipitate confirms the presence of Al$^{3+}$. Since aluminon is a soluble red dye, the solution must be
centrifuged to determine the color of the precipitate. Whether aluminum is present or not, the supernatant liquid will have a red color. The formation of red precipitate indicates aluminum.

**Chromium(III) Ion**: The test for chromium involves reduction of dichromate ion by hydrogen peroxide in acidic solution to give the blue CrO$_5$ species. CrO$_5$ is unstable and the blue color fades rapidly.

\[
\text{Cr}_2\text{O}_7^{2-}(aq) + 4\text{H}_2\text{O}_2(aq) + 2\text{H}^+(aq) \rightarrow 2\text{CrO}_5(aq) \text{ (blue)} + 5\text{H}_2\text{O(l)}
\]

\[
\text{CrO}_5(aq) + 6\text{H}^+(aq) \rightarrow \text{Cr}^{3+}(aq) + \text{O}_2(aq) + 3\text{H}_2\text{O(l)}
\]

The fleeting appearance of a blue color confirms Cr$^{3+}$.

**Waste Disposal**: In all laboratory procedures, proper disposal of waste is an important environmental and legal issue. CCRI's policy is not to pour *any* chemicals down the drain. During metal cation analysis, all waste must go into the heavy metal waste collection container. Chromium, for instance, has a 0.5 ppm discharge limit; one act of careless disposal could exceed this value and put CCRI at risk for a sewer authority citation. The situation is *much* more critical with mercury has a discharge limit of only 30 parts per billion. Extreme care is needed to keep even the smallest trace of this dangerous pollutant out of any sewer system.

Waste Disposal: Collect all solid and liquid waste and the rinse water from your glassware in a marked beaker at your bench, even if you are not sure if disposal of the metal you are using is regulated or not. At the end of the lab empty the beaker contents and rinse the beaker into the container marked "Heavy Metal Waste". Do not wash anything in the sink until all chemical residues have been transferred to the heavy metal waste collection container.

**Experimental**: Place 20 drops of the known and unknown solutions saved from the Group A analysis into separate test tubes. Carry out the same tests on the known and unknown solutions and record your observations.

Add 6M HCl to each solution with stirring until it is acidic to litmus. Add 1M NaHCO$_3$ dropwise (fizzing will occur) until the solutions test neutral with litmus paper. Centrifuge each test tube for two minutes and transfer the supernatant liquid from each (containing Al$^{3+}$ and Cr$^{3+}$ cations) to separate, clean test tubes. Test each precipitate for Sn$^{2+}$ and then test the supernatant liquid from each test tube for aluminum and chromium.

**Confirmation of Sn$^{4+}$ Ion**: Wash the precipitate from above with 20 drops of distilled water. Centrifuge and discard the wash liquid. Dissolve the precipitate in 8 drops of 6M HCl and add a small piece of aluminum wire. If necessary, add another drop or two of 6M HCl to dissolve any excess aluminum metal. Afterwards, add one drop of 0.1M HgCl$_2$ and note if a white solid forms. A white solid confirms the presence of Sn$^{2+}$,

**Confirmation of Al$^{3+}$ Ion**: Place four drops of the supernatant liquid saved from the above procedure into a clean test tube. Add 4 drops of 6M HCl (fizzing will occur). Add 4 drops of aluminon solution and then add 6M NH$_3$ until the solution is basic to litmus. Centrifuge for one minute and decant. A red precipitate confirms the presence of Al$^{3+}$. 
Confirmation of Cr\textsuperscript{3+} Ion: Add 2 drops of 3% H\textsubscript{2}O\textsubscript{2} to the remaining supernatant liquid. Add 6M HCl (fizzing will occur) until the solution tests acidic to litmus. Observe the solution for a few minutes. The appearance of a flash of blue color confirms the presence of Cr\textsuperscript{3+}.

| Name:_______________________________ | Section No:_____ |

**Qualitative Analysis of Group B Cations**  
**Data Page for Known Solution**

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Qualitative Analysis of Group B Cations
Data Page for Unknown Solution

Unknown No.______________

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1. What Group B cation(s) are present in your unknown?

_________  _______  _______  (15 points)

2. Draw a flow diagram showing the steps and products you found in the analysis of your unknown. (5 points)
General Chemistry II Lab

Prestudy for Experiment #3 Inorganic Qualitative Analysis

Separation and Characterization of Group B Cations

An unknown solution containing only Group B cations is treated as described in the lab procedure. At each stage below, state which cations may be present, which are confirmed present, and which are absent.

(a) Without any treatment, the solution has a yellow color. (5 points)

(b) The liquid from step (a) is treated with HCl and NaHCO₃. A white precipitate and orange liquid results. (5 points)

(e) The precipitate from step (b) is dissolved in HCl and treated with aluminum metal and then Hg²⁺. A white solid forms. (5 points)

(d) The supernatant liquid from step (b) is divided into two portions. One part is treated with HCl, aluminon, and NH₃ giving a clear red solution with no precipitate. The second part is treated with 3% H₂O₂ and HCl. The solution turns dark blue and quickly fades to colorless. (5 points)