Qualitative Analysis

Anion Analysis:

1. Prepare a mixture of the four anions by adding approximately 1 mL of the following 0.10 mol/L solutions to a clean test tube: Na_2SO_4, Na_2CO_3, NaCl and NaI. A sample flow chart can be found below. Fill in the colours of the solutions and precipitates as they are observed.
2. Pour 2 mL of your anion solution into another clean test tube and add 20 drops of 0.20 mol/L $Ba(NO_3)_2$. Record your observations.
3. Centrifuge for 1 minute and then test the solution for complete precipitation by adding 2 more drops of the $Ba(NO_3)_2$ solution. If more precipitate forms, centrifuge again and then go onto step 4. If no precipitate forms, go on to procedure 4 right away.
4. Decant the supernatant liquid and save it for step 7.
5. Wash the precipitate with 10 drops of distilled water then centrifuge. Decant and discard the wash solution.
6. To the precipitate from procedure 3, add 10 drops of 6.0 mol/L HNO_3 (aq).
   Stir with a glass rod and observe.
   If bubbling occurs and some of the precipitate disappears, this **confirms the presence of CO_3^{2-}** as $BaCO_3$.
   If some of the precipitate remains, this **confirms the presence of SO_4^{2-} as $BaSO_4$.** If **all** of the precipitate disappears or **none** of it disappears, what does this tell you?
7. To the solution from step 4, add 20 drops of 0.10 mol/L $AgNO_3$. Record your observations.
8. Centrifuge, decant and discard the supernatant liquid. Wash the precipitate with 10 drops of distilled water. Decant and discard the wash solution.
9. Add 20 drops of 6.0 mol/L NH_4OH solution to the precipitate and stir with a glass rod.
10. Centrifuge and separate the solution from the precipitate and set it aside for procedure 11. Note the colour of the precipitate. Finding a precipitate of this colour at this point in the procedure **confirms the presence of I^{1-} as AgI**.
11. To the solution from procedure 10, add 10 drops of 6.0 mol/L HNO_3 (aq). Note the colour of the precipitate. Finding a precipitate of this colour at this point in the procedure **confirms the presence of Cl^{1-} as AgCl.**

Cation Analysis:

Make up a test solution by adding 20 drops of each of the available 0.25mol/L nitrate solutions of Fe^{3+}, Cr^{3+}, Ni^{2+} and Co^{2+}

Part A: The Iron Group, Fe^{3+} and Cr^{3+}

1. Add 2.0 mL of 6.0 mol/L NH_4OH to your test solution and stir thoroughly. The dark precipitate that forms is a mixture of $Fe(OH)_3$ and $Cr(OH)_3$.

2. Centrifuge then test the solution for complete precipitation. When you are sure that all the $Fe(OH)_3$ and $Cr(OH)_3$ has been removed, decant the solution and set it aside for procedure 11. Wash the precipitate with a few drops of distilled water, re-centrifuge and discard the wash solution.

3. Dissolve the precipitate from procedure 3 by adding 6.0 mol/L HCl drop by drop and stirring with a glass rod.

4. Make the solution strongly basic to litmus by adding 6.0 mol/L NaOH until litmus turns blue then add three more drops. A **rust-coloured precipitate of $Fe(OH)_3$** should appear.

5. Using a test tube rack, carry the test tube to the fume hood and carefully add 20 drops (one or two at a time) of 30% H_2O_2 (hydrogen peroxide) solution. (If too much peroxide is added at one time, the solution may foam up and spill down the outside of the test tube creating a very dangerous situation.)

6. Return to your lab station and gently heat the test tube in a hot water bath. Once again there is a danger the solution may foam up and spill down the outside of the test tube. If this starts to happen, remove the tube from the water bath until the bubbling subsides. This warming decomposes the remaining hydrogen peroxide. It takes about 15 minutes. While you are waiting, you could go on to procedure 11.

7. Centrifuge and decant the solution into another test tube and set it aside for procedure 10. (The solution should be yellow, indicating the presence of the CrO_4^{2-} ion.)

8. Dissolve the precipitate from procedure 8 by adding 6.0 mol/L HCl a few drops at a time and stirring. Add 30 drops of 0.1 mol/L KSCN solution. The appearance of a **deep blood-red coloured solution of $Fe(SCN)^{2+}_{(aq)}$ confirms the presence of the Fe^{3+}**

9. Add 5 drops of 0.1 mol/L $BaCl_2$ solution to the solution from procedure 8. A **yellow precipitate of $BaCrO_4$ confirms the presence of Cr^{3+} in the original solution.**

Part B: The Nickel Group, Ni^{2+} and Co^{2+}

11. Divide the solution from procedure 3 into two separate test tubes. Use one portion for procedure 12, and one for procedure 13.

12. To the first portion of the solution from procedure 3, add 10 to 15 drops of dimethyl glyoxime and warm in a water bath. **A strawberry-red precipitate of $NiC_8H_{14}N_4O_4$ confirms the presence of the Ni^{2+} ion** in the original solution.

13. Acidify the second portion of the solution from procedure 3 by adding 6.0mol/L HNO_3 one drop at a time. Add 1.0mL of 6.0mol/L of KNO_2 solution then warm and let stand for 15 minutes in a hot water bath. Beware that brown fumes of $NO_2(g)$ may be emitted. Make sure your hot plate is close to a fume vent.

14. After heating the tube for 15 minutes, cool and place it in the centrifuge**. A yellow precipitate of $K_3Co(NO_2)_6$ confirms the presence of Co^{2+} in the original solution**.

At this point, you are ready to proceed with the unknown identification. Show your completed flowchart to the teacher and obtain a numbered test tube which contains a combination of 3 ions from the 8 you have already analyzed.

Part A: Anion Analysis: SO_4^{2-}, CO_3^{2-}, Cl^{1-} and I^{1-}.

1. Measure out approximately 1 mL of your unknown solution into a clean test tube. Add 5 drops of 6.0 mol/L NH_4OH and stir. A precipitate of iron and nickel group may appear.
2. Centrifuge and decant the solution into a clean test tube. Discard the precipitate. Add 20 drops of 0.20 mol/L $Ba(NO_3)_2$ and stir.
3. Centrifuge for 1 minute then test the solution for complete precipitation by adding 2 more drops of the $Ba(NO_3)_2$ solution. If more precipitate forms, centrifuge again then go onto procedure 4. If no precipitate forms go on to procedure 4 right away.
4. Decant the supernatant liquid and save it for procedure 7.
5. Wash the precipitate with 10 drops of distilled water then centrifuge. Decant and discard the wash solution.
6. To the precipitate from procedure 3, add 10 drops of 6.0 mol/L HNO_3. Stir with a glass rod and observe.
   If bubbles form and the _____ coloured precipitate disappears, this **confirms the presence of CO_3^{2-} in your solution.**
   If none of the _____ coloured precipitate disappears and no bubbles form, this **confirms the presence of SO_4^{2-} in your solution.**
   What should you observe if both anions are present?
   What should you observe if neither anions are present?
7. To the solution from procedure 4, add 20 drops of 0.10 mol/L $AgNO_3$. If a precipitate forms, proceed with step 8; if no precipitate forms, what does this tell you?
8. Centrifuge, decant and discard the supernatant liquid. Wash the precipitate with 10 drops of distilled water. Decant and discard the wash solution.
9. Add 20 drops of 6.0 mol/L NH_4OH solution to the precipitate and stir with a glass rod.
10. Centrifuge and separate the solution from the remaining precipitate (if there is one) and set it aside for procedure 11. A _____ coloured precipitate remaining at this point in the procedure **confirms the presence of I^{1-}** in your solution.
11. To the solution from procedure 10, add 10 drops of 6.0 mol/L HNO_3. If a _____ coloured precipitate forms, this **confirms the presence of Cl^{1-}** in your solution.
12. Discard the remaining solution and precipitates from this part of the procedure.

Part B: Cation Analysis: The Iron Group: Fe^{3+} and Cr^{3+}

1. To the solution from Part A: Procedure 2, add 2 mL of 6.0 mol/L NH_4OH to your test solution and stir thoroughly. If a precipitate forms, what does this tell you? What does the colour of the precipitate tell you? If no precipitate forms, what does this tell you?
2. Centrifuge and then test the solution for complete precipitation. When you are sure that all the Iron group ions have been removed, decant the solution and set it aside for procedure 11.
3. Wash the precipitate with a few drops of distilled water, re-centrifuge and discard the wash solution.
4. Dissolve the precipitate from procedure 3 by adding 6.0 mol/L HCl drop by drop and stir with a glass rod.
5. Make strongly basic to red litmus by adding 6.0 mol/L NaOH until the litmus turns blue then add three more drops.
6. Using a test tube rack, carry the test tube to the fume hood and carefully add 209 drops (one or two at a time) of 30% H_2O_2 (hydrogen peroxide) solution. (Remember: If too much peroxide is added at one time, the solution may foam up and spill down the outside of the test tube creating a very dangerous situation.)
7. Return to your lab station and gently heat the test tube in a hot water bath. Once again there is a danger the solution may foam up and spill down the outside of the test tube. If this starts to happen, remove the tube from the water bath until the bubbling subsides. This warming decomposes the remaining hydrogen peroxide. It takes about 15 minutes. While you are waiting, you could go on to procedure 11.
8. Centrifuge and decant the solution into another test tube and set it aside for procedure 10. If there is a precipitate, treat it by procedure 9. If there is no precipitate, what does this tell you?

9. Dissolve the precipitate from procedure 8 by adding 6.0 mol/L HCl a few drops at a time and stirring. Add 30 drops of 0.10 mol/LKSCN solution. The appearance of a _____ coloured solution **confirms the presence of the Fe^{3+} in your solution.**

10. Add 5 drops of 0.10 mol/L $BaCl_2$ to the solution from procedure 8. A _____coloured precipitate of $BaCrO_4$ **confirms the presence of Cr^{3+} in your solution.**

Part B: Cation Analysis: Ni^{2+} and Co^{2+}

11. Divide the solution from procedure 3 into two separate test tubes. Use one portion for procedure 12 and one for procedure 13.
12. To the first portion of the solution from procedure 3, add 10 to 15 drops of dimethyl glyoxime and warm in a water bath. **A strawberry-red precipitate of $NiC_8H_{14}N_4O_4$ confirms the presence of the Ni^{2+} ion** in the original solution.
13. Acidify the second portion of the solution from procedure 3 by adding 6.0mol/L HNO_3 one drop at a time. Add 1.0mL of 6.0mol/L of KNO_2 solution then warm and let stand for 15 minutes in a hot water bath. Beware that brown fumes of $NO_2(g)$ may be emitted. Make sure your hot plate is close to a fume vent.
14. After heating the tube for 15 minutes, cool and place it in the centrifuge**. A yellow precipitate of $K_3Co(NO_2)_6$ confirms the presence of Co^{2+} in the original solution**.