CH 223 Spring 2025: **“Qualitative Analysis of Group III Cations”** *(in class)* ***Lab:*** *Instructions*

Note: **This is the lab for section 01 and H1 of CH 223 only.**

- *If you are taking section W1 of CH 223*, please use this link: http://mhchem.org/q/9b.htm

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Step One:

Get a printed copy of this lab! You will need a printed (hard copy) version of pages Ia-9-2 through Ia-9-5 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Bring the printed copy of the lab with you on Monday, June 2 (section 01) *or* **Wednesday, June 4 (section H1).** During lab in room AC 2507, you will use these sheets (with the valuable instructions!) to gather data, all of which will be recorded in the printed pages below.

Step Three:

Complete the lab work, then **turn it in** (page Ia-9-5 *only* to avoid a point penalty) **at the** ***END OF LAB*** **to the instructor on Monday, June 2 (section 01)** *or* **Wednesday, June 4 (section H1.)**

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Qualitative Analysis Of Group III Cations

The four group III cations in this lab are $\mathbf{Cr^{+3}}$, $\mathbf{Al^{+3}}$, $\mathbf{Fe^{+3}}$, and $\mathbf{Ni^{+2}}$. The first step in analysis involves separating the ions into two subgroups by treating the solution with NaOH and NaOCl. The hypochlorite ion oxidizes Cr(III) to a higher, more stable oxidation state (namely Cr(VI)) which is soluble:

$$2\ Cr^{+3}(aq) + 3\ OCl^{-1}(aq) + 10\ OH^{-1}(aq) \rightarrow 2\ CrO_4^{-2}(aq) + 3\ Cl^{-1}(aq) + 5\ H_2O(l)$$

In addition, Al^{+3} forms a soluble hydroxo-complex ion in the presence of excess hydroxide:

$$Al^{+3}(aq) + 4\ OH^{-1}(aq) \rightarrow Al(OH)_4^{-1}(aq)$$

In contrast, Ni^{+2} and Fe^{+3} do not readily form hydroxo-complexes and are not oxidized by hypochlorite. They form insoluble hydroxides under these conditions:

$$Ni^{+2}(aq) + 2\ OH^{-1}(aq) \rightarrow Ni(OH)_2(\text{green solid})$$

$$Fe^{+3}(aq) + 3\ OH^{-1}(aq) \rightarrow Fe(OH)_3(\text{red solid})$$

To separate aluminum from chromium, the solution containing CrO_4^{-2} and $Al(OH)_4^-$ is acidified to destroy the hydroxo-complex:

$$Al(OH)_4^{-1}(aq) + 4\ H^+(aq) \rightarrow Al^{+3}(aq) + 4\ H_2O(l)$$

Treatment with aqueous ammonia gives a gelatinous **white** precipitate of aluminum hydroxide. The concentration of hydroxide in ammonia is too low to form the hydroxo-complex:

$$Al^{+3}(aq) + 3\ NH_3(aq) + 3\ H_2O(l) \rightarrow 3\ NH_4^+(aq) + Al(OH)_3(\text{white solid})$$

The chromate ion remains in solution. It can be tested and confirmed by precipitation as yellow $BaCrO_4$:

$$Ba^{+2}(aq) + CrO_4^{-2}(aq) \rightarrow BaCrO_4(\text{yellow solid})$$

The $BaCrO_4$ precipitate dissolves in acid. The solution is then treated with H_2O_2 to produce a **deep blue** color due to the presence of a peroxo-compound, probably CrO_5:

$$2\ BaCrO_4(s) + 4\ H^+(aq) + 4\ H_2O_2(aq) \rightarrow 2\ Ba^{+2}(aq) + 6\ H_2O(l) + 2\ CrO_5(\text{blue, aq})$$

The mixed precipitate of $Ni(OH)_2$ and $Fe(OH)_3$ can be dissolved by adding a strong acid. The Ni^{+2} and Fe^{+3} ions can be separated by adding ammonia. Ni^{+2} is converted to the deep-blue complex $Ni(NH_3)_6^{+2}$ which stays in solution. The Fe^{+3} ion does not readily form a complex and re-precipitates as $Fe(OH)_3$:

$$Ni^{+2}(aq) + 6\ NH_3(aq) \rightarrow Ni(NH_3)_6^{+2}(\text{blue, aq})$$

$$Fe^{+3}(aq) + 3\ NH_3(aq) + 3\ H_2O(l) \rightarrow 3\ NH_4^+(aq) + Fe(OH)_3(\text{red solid})$$

Confirm the presence of Ni^{+2} by adding dimethylglyoxime (DMG), $C_4H_8N_2O_2$, to give a **deep rose** precipitate:

$$Ni^{+2}(aq) + 2\ C_4H_8N_2O_2(aq) \rightarrow 2\ H^+(aq) + Ni(C_4H_7N_2O_2)_2(\text{red solid})$$

Confirm the presence of Fe^{+3} by dissolving $Fe(OH)_3$ in a strong acid and adding KSCN to form a **blood-red** $FeSCN^{+2}$ complex ion:

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

<u>PROCEDURE</u>: ***Safety glasses required for everyone***, *all the time - no open toed shoes or sandals, no shorts or mid-riff showing shirts.* ***This can be a longer lab***, *so come prepared and organized to maximize your lab experience.*

Step 1: Preparation: If you are working on the analysis of only group III ions, prepare a known solution by mixing 5-6 drops each of 0.1 M solutions of Fe^{+3}, Al^{+3}, Cr^{+3}, and Ni^{+2} in a 30 mL beaker. Also obtain an unknown to analyze at the same time for the presence of group III cations and use about 20-24 drops in your analysis.

Step 2: Oxidation of Cr(III) to Cr(VI) and Separation of Insoluble Hydroxides: Add 1 mL of 6 M NaOH to the solution in a 30 mL beaker. Boil very gently for 1 minute while stirring. Remove heat and slowly add dropwise 1 mL of 1 M NaClO. Swirl the beaker for 30 seconds, using tongs if necessary. Boil the mixture for 1 minute. Centrifuge. Transfer the supernatant (which contains the aluminum and chromate) to a separate tube. Wash the precipitate (which contains iron and nickel hydroxides) with 2 mL water and 0.5 mL of 6 M NaOH: stir, centrifuge, decant and discard the wash. Add 1 mL of water and 1 mL of 6 M HNO_3 to the solid and put it aside until step 6.

Step 3: Separation of Al from Cr: Acidify the solution from step 2 by adding 6 M acetic acid in **2.0 mL increments** until, after stirring, the mixture is definitely acidic to litmus (it might take up to 30 mL or more.) If necessary, transfer the solution to a 30 mL beaker and boil it to reduce its volume to about 3 mL. Pour the solution into a test tube. Add 6 M NH_3 in 5 drop increments until the solution is basic to litmus; and then add 0.5 mL excess. Stir the mixture for one minute to bring the system to equilibrium. If aluminum is present, a light, translucent gelatinous white precipitate of $Al(OH)_3$ may be floating in the clear to yellow solution. Centrifuge and transfer the liquid (which contains CrO_4^{-2}) to another test tube.

Step 4: Confirmation of the Presence of Aluminum: Wash the precipitate from step 3 with 3 mL water, warming the test tube in the water bath and stirring well. Centrifuge and discard the wash. Dissolve the precipitate in 2 drops of 6 M CH_3CO_2H (no more, no less!). Add 3 mL of water and 1-2 drops of aluminon. Stir. If Al^{+3} is present, the solution will turn a lovely rose-pink color due to the presence of a very fine red or pink precipitate.

Step 5: Confirmation of the Presence of Chromium: A yellow liquid from step 3 *suggests* but does not confirm the presence of chromium. To the solution add 0.5 mL of 1.0 M $BaCl_2$. In the presence of chromium a finely divided yellow precipitate of $BaCrO_4$ appears. Put the test tube in boiling water for two minutes. Centrifuge and discard the liquid. Wash the solid with 2 mL of water, centrifuge and discard the wash. To the solid add 0.5 mL of 6 M HNO_3. Stir to dissolve $BaCrO_4$. Add 1 mL of water; stir the orange solution. Add 2 drops of 3% H_2O_2. A deep blue solution, which may fade rapidly, confirms the presence of chromium.

Step 6: Separation of Iron and Nickel: Returning to the precipitate from step 2, stir to dissolve the solid in the HNO_3. If necessary, warm the test tube in the water bath to completely dissolve the solid. Add 6 M NH_3 until the solution is basic to litmus. At this point, iron will precipitate as brown $Fe(OH)_3$. Add 1 mL more of the NH_3 and stir to bring the nickel into solution as the $Ni(NH_3)_6^{+2}$ ion. Centrifuge and decant the liquid into a test tube. Save the precipitate for step 8 to test for the presence of iron.

Step 7: Confirmation of the Presence of Nickel: If the solution from step 6 is blue, nickel may be present. To the solution add 0.5 mL dimethylglyoxime (DMG) reagent. Formation of a rose-red precipitate proves the presence of nickel.

Step 8: Confirmation of the Presence of Iron: Dissolve the precipitate from step 6 in 0.5 mL of 6 M HCl. Add 2 mL water and stir. Add 2 drops of 1.0 M KSCN. Iron is present if a deep red solution of $FeSCN^{+2}$ is formed.

Step 9: Cleanup! Please rinse all glassware and equipment prior to leaving lab. Return cleaned centrifuge tubes to the container without masking tape.

Make sure you include your unknown number or letter!

Answers to the postlab questions can mostly be found on the main page of this lab, hint, hint ☺.

Qualitative Analysis of Group III Cations Lab - *Worksheet*

YOUR NAME: ________________________ ***LAB PARTNER(s):*** __________________

Unknown Number: ___________

Circle *either* yes or no for each metal cation in your unknown.

Fe^{3+}:	yes	no
Ni^{2+}:	yes	no
Cr^{3+}:	yes	no
Al^{3+}:	yes	no

Postlab Questions:

1. Write balanced net ionic equations for the following reactions:
   a. Dissolving $Fe(OH)_3$ in nitric acid

   b. Oxidation of Cr(III) by hypochlorite ion in base.

   c. The confirmatory test for Ni^{+2}. (Step 7)

   d. The confirmatory test for Fe^{+3}. (Step 8)

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