Group B Cation Analysis

Inorganic Qualitative Analysis

<u>Introduction</u>: 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 HC1, taking care to note the appearance a 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.

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

$$Sn(OH)_6^{-1}(aq) + 6H^{+1}(aq) + 6Cl^{-1}(aq) \rightarrow SnC1_6^{-2}(aq) + 6H_2O(l)$$

$$2CrO_4^{-2}(aq) \text{ (yellow)} + 2H^{+1}(aq) \rightarrow Cr_2O_7^{-2}(aq) \text{ (orange)} + H_2O(l)$$

This solution is next treated with bicarbonate to neutralize the acid. Tin(IV) ion precipitates as SnO₂, while dichromate and aluminum ions are unaffected.

$$SnC1_6^{-2}(aq) + 2H_2O(1) + 4NH_3(aq) \rightarrow SnO_2(s) + 4NH_4^{+1}(aq) + 6Cl^{-1}(aq)$$

Tin (IV) Ion: Solid SnO_2 is treated with HC1 to dissolve the precipitate. Aluminum metal is then added to convert the solid tin(II) oxide to $SnCl_4^{-2}$.

$$SnO_{2}(s) + 4H^{+l}(aq) + 6Cl^{-l}(aq) \rightarrow SnC1_{6}^{-2}(aq) + 2H_{2}O(l)$$
$$3SnC1_{6}^{-2}(aq) + 2A1(s) \rightarrow 3SnC1_{4}^{-2}(aq) + 2Al^{+3}(aq) + 6Cl^{-1}(aq)$$

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

$$2Al(s) + 6H^{+l}(aq) \rightarrow 2Al^{+3}(aq) + 3H_2(g)$$

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_2C1_2 , confirming Sn^{+2} .

$$SnC1_4^{-2}(aq) + 2Hg^{+2}(aq) + 4Cl^{-1}(aq) \rightarrow Hg_2C1_2(s)$$
 (white) $+ SnC1_6^{-2}(aq)$

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 CrO₅ species. CrO₅ is unstable and the blue color fades rapidly.

$$Cr_2O_7^{-2}(aq) + 4H_2O_2(aq) + 2H^{+1}(aq) \rightarrow 2CrO_5(aq) \text{ (blue)} + 5H_2O(1)$$

$$CrO_5(aq) + 6H^{+1}(aq) \rightarrow Cr^{+3}(aq) + O_2(aq) + 3H_2O(1)$$

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.

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 NaHCO₃ 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 HC1 and add a small piece of aluminum wire. If necessary, add another drop or two of 6M HC1 to dissolve any excess aluminum metal. Afterwards, add one drop of $0.1M \, \text{HgCl}_2$ 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 HC1 (fizzing will occur). Add 4 drops of aluminon solution and then add 6M NH₃ until the solution is basic to litmus. Centrifuge for one minute and decant. A red **precipitate** confirms the presence of Al⁺³. Remember a clear red solution indicates no Al³⁺.

Confirmation of Cr^{+3} Ion: Add 2 drops of 3% H_2O_2 to the remaining supernatant liquid. Add 6M HC1 (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^{+3} . Sometimes the blue blue color will not appear, especially if the hydrogen peroxide is not fresh. If the solution is yellow or yellow-orange Cr^{3+} is present.

Name:	
	Qualitative Analysis of Group B Cations

Data Page for Known Solution

	Conclusion

Name:		
Q	Qualitative Analysis of Group B Cati	ons
	Data Page for Unknown Solution	
nknown No		
Test	Result	Conclusion

Na	me:Results of Group B Cations
Un	known No.
1.	What Group B cation(s) are present in your unknown?

Name:				
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des	The yellow solution remaining after Group A separation and containing possible Group B cations is treated as described in the lab procedure. At each stage below indicate what the following tests tell you about which cations may be present, which are confirmed present, which are definitely absent, and why.			
(a)	Without any treatment, the solution has a yellow color.			
(b)	The solution from step (a) is treated with HC1 and NaHCO ₃ , centrifuged, and separated to give the following to give the following results.			
	1. A white precipitate is obtained.			
	2. An orange solution is obtained.			
(c)	The precipitate from step (b) is dissolved in HC1 and treated with aluminum metal and then Hg^{+2} to give a white solid.			
(d)				
	2. The second portion is treated with $3\%~H_2O_2$ and HC1. The solution turns dark blue and quickly fades to colorless			