

## QUALITATIVE ANALYSIS of GROUP III CATIONS

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



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



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



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



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:



The chromate ion remains in solution. It can be tested and confirmed by precipitation as yellow  $\text{BaCrO}_4$ :



The  $\text{BaCrO}_4$  precipitate dissolves in acid. The solution is then treated with  $\text{H}_2\text{O}_2$  to produce a **deep blue** color due to the presence of a peroxo-compound, probably  $\text{CrO}_5$ :



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



Confirm the presence of  $\text{Ni}^{2+}$  by adding dimethylglyoxime (DMG),  $\text{C}_4\text{H}_8\text{N}_2\text{O}_2$ , to give a **deep rose** precipitate:



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



# Qualitative Analysis Of Group Iii Cations

**George Wallace Sears**



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