5.3: Procedure, flowchart, and datasheets for separation and confirmation of group III cations

Table 1. List of chemicals and their hazards*

Chemical	Hazard
0.1M ammonium chloride (NH_4Cl)	Toxic and irritant
0.1M Chromium(III) chloride	Toxic and irritant
0.1M Iron(III) chloride	Toxic and corrosive
0.1M Nickel(II) chloride	Toxic, irritant, and suspected carcinogen

- *Hazards of 6M ammonia, 6M hydrochloric acid, 6M nitric acid, 3% hydrogen peroxide, and 1M thioacetamide are listed in chapter 2 in the commonly used reagent section. **Caution!** Used heavy metal ion solutions are disposed of in a labeled metal waste disposal container, do not drain these solutions down the drain.

Caution

- Used heavy metal ion solutions or precipitates are disposed of in a labeled metal waste disposal container, do not drain these solutions down the drain or in the regular trash.

Procedure for the analyses of group III cations

1. Take 15 drops of the fresh test solution if group I and group II cations are not present in the sample or **take the supernatant of step 1 of group II cations analysis**. Add 2 drops of 6M HCl. Then add 6M NH_3 drop by drop while stirring till the solution turns basic. Use red litmus paper to test -when it turns blue the solution is basic. Add 5 more drops of 6M NH_3 after the solution becomes alkaline to make NH_3/NH_4^+ buffer. Record the observations in the datasheet.
2. Add 10 drops of 1M thioacetamide to the solution from step 1, stir, and heat in a boiling water bath for 10 min. Then centrifuge for 2 min and decant. **Keep the supernatant for group IV** cations analysis and **keep the precipitate** for group III cations. Record the observations in the datasheet.
3. Wash the group III precipitates from step 2 by re-suspending in 15 drops of 0.1M NH_4Cl. Then centrifuge, decant, and discard the supernatant that is just the washing liquid. Re-suspend the precipitates in 10 drops of 6M HCl, heat for 2 min in a boiling water bath, centrifuge for two minutes, and **keep the supernatant** that may contain Fe^{2+} and/or Cr^{3+} and **keep the precipitate**, if there is any. Record the observations in the datasheet.
4. Wash the precipitate of step 3 by re-suspending it in 15 drops of distilled water. Then centrifuge for 1 min, decant and discard the supernatant that is just the washing liquid. Re-suspend the precipitate after adding 4 drops of 6M HCl and 6 drops of 6M HNO_3 (i.e., aqua regia). Heat the suspension in a boiling water bath for 2 min, then centrifuge for 2 min, decant, and discard the precipitate which is solid sulfur that contains no ions in it, but **keep the supernatant** for nickel analysis.
5. Use the cotton-plug technique to aspirate clear supernatant if it is not already a clear solution. Add 6M NH_3 to the clear supernatant drop by drop till the solution turns alkaline. Use red litmus paper to test -when it turns blue the solution is alkaline. If the solution turns turbid at this stage, centrifuge for 1 min, decant and discard the precipitate, but **keep the supernatant**.
6. Add 5 drops of dimethylglyoxime to the clear solution of step 5, stir, and leave for a minute. If a bright red precipitate is formed at this stage, it confirms Ni^{2+} is present in the test sample. Discard the mixture in the metal waste container and record the observations in the datasheet.
7. Inspect the supernatant of step 3, if it is not clear make it clear using the cotton plug technique. Add 6M NH_3 drop-by-drop to the clear supernatant while stirring till the mixture until it turns alkaline and has pH in the range of 9 to 10. Use a pH paper (not a litmus paper) to determine the pH. Add 5 drops of 3% H_2O_2 to the alkaline solution, stir, and leave for half-min. Heat the mixture in a boiling water bath to destroy excess H_2O_2 till the oxygen gas bubbles stop evolving from the solution. It may take about 3 min or more. Centrifuge for 1 min and test again for pH with a pH paper -if pH is less than 9,

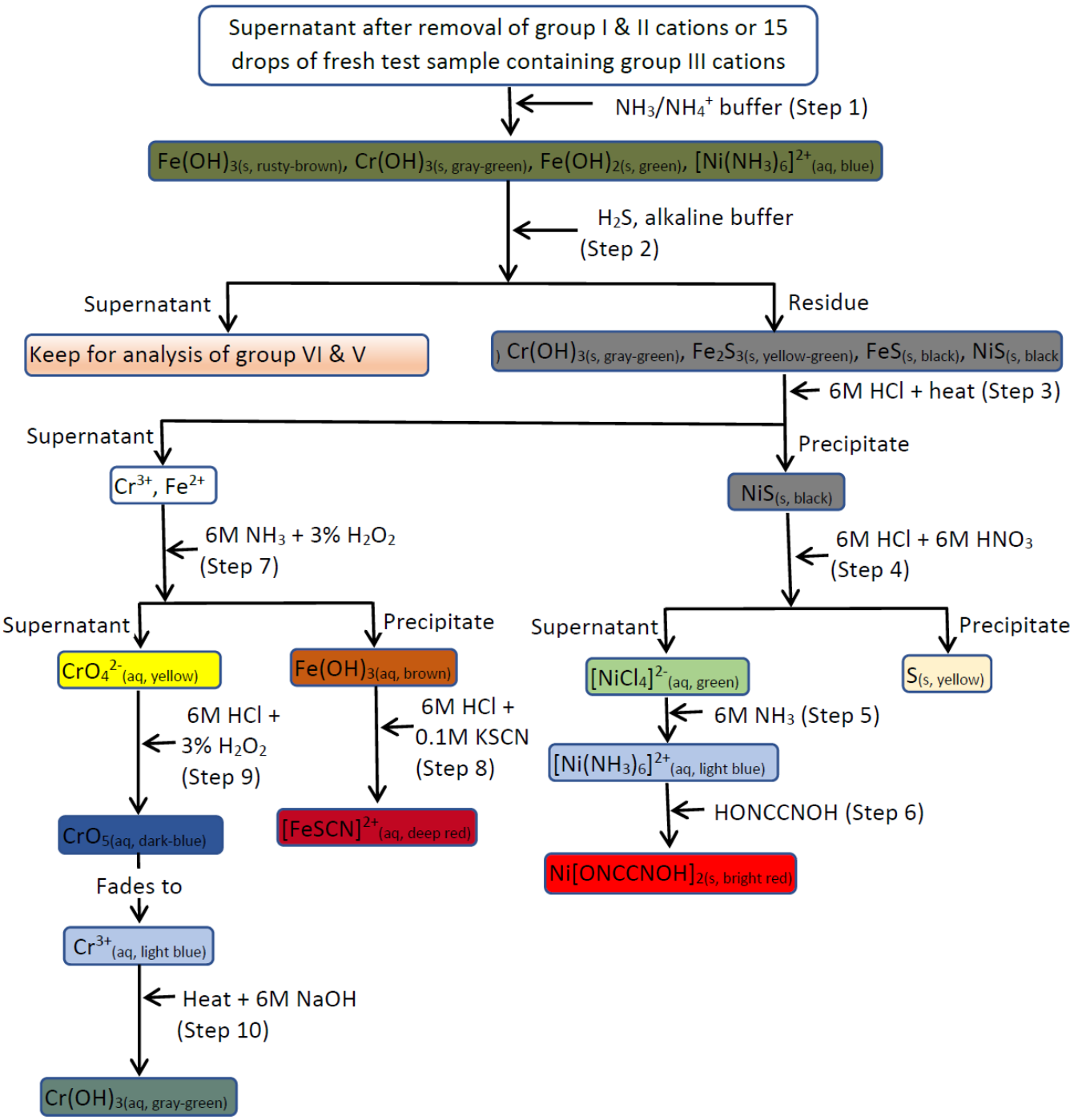
repeat this step 7 from the beginning, otherwise decant and **keep the supernatant** for analysis of chromium ions and **keep the precipitate**, there is any, for analysis of iron ions. Record the observations in the datasheet.

8. Dissolve the precipitate from step 7 in 5 drops of 6M HCl under stirring. Add 5 drops of distilled water to the solution followed by 5 drops of 0.1M potassium thiocyanate ($KSCN$) and stir to mix. If the solution color changes to deep red, it confirms iron ions are present in the test sample. Discard the mixture in a metal waste container, and record the observations in the datasheet.
9. To the supernatant of step 7 add 6M HNO_3 drop by drop till the solution becomes acidic with pH ~3. Use pH paper(not litmus paper) to determine the pH. Then add 1 drop of 3% H_2O_2, mix and leave for half-min. Then heat in boiling water bath to destroy excess H_2O_2 till oxygen bubbles stop forming in the mixture. It may take about 3 min or more. Cool the mixture by placing it in a room temperature water bath.
10. Add 6M $NaOH$ drop by drop to the solution of step 9 at room temperature till the solution is basic, i.e., turn red litmus paper to blue. The formation of gray-green precipitate at this stage confirms Cr^{3+} is present in the test sample. Caution: H_2O_2 decomposes slower in the acidic medium than in the basic medium. If the solution turns dark blue or yellow, it indicates chromium is present as CrO_5 or CrO_4^{2-} and H_2O_2 was not destroyed completely. In this case, add 6M HNO_3 drop by drop with stirring till the color fades away. Then repeat the addition of 6M NaOH till the solution turns basic and observe. The formation of gray-green precipitate at this stage confirms Cr^{3+} is present in the test sample. Discard the mixture in a metal waster container and record the observations in the datasheet.

Datasheets filling instructions for group III cations

1. Step number refers to the corresponding step number in the procedure sub-section.
2. In “the expected chemical reaction and expected observations column”, write an overall net ionic equation of the reaction that will happen if the ion being processed in the step was present, write the expected color change of the solution, the expected precipitate formed and its expected color, etc.
3. In the “the actual observations and conclusion” column write the color change, the precipitate formed and its color, etc. that is actually observed as evidence, and state the specific ion as present or absent.
4. In “the overall conclusion” row write one by one symbol of the ions being tested with a statement “present” or “absent” followed by evidence/s to support your conclusion.

Group-III Cations Analysis Flow Chart



Data sheet for known Group III cations (Cr^{3+}, Fe^{3+}, Fe^{2+}, and Ni^{2+}) analysis

Students Name: _____ Group partners: _____ Date: _____

Step# Net ionic equation, and observations from the expected reaction	Actual observations and conclusion
Step 1	
Step 2	
Step 3	
Step 4	
Step 6	
Step 8	
Step 9	
Step 10	
Overall conclusion	

Datasheet for unknown sample#_____ of Group III cations (Cr^{3+}, Fe^{3+}, Fe^{2+}, and Ni^{2+}) analysis

Students Name: _____ Group partners: _____ Date: _____

Step# Net ionic equation, and observations from the expected reaction	Actual observations and conclusion
Step 1	
Step 2	
Step 3	
Step 4	
Step 6	
Step 8	
Step 9	
Step 10	
Overall conclusion	