4.3: Procedure, flowchart, and datasheets for separation and confirmation of group II cations

Table 1: List of chemicals and their hazards*

Chemical	Hazard
0.1M ammonium chloride (NH_4Cl)	Toxic and irritant
0.1M bismuth nitrate in 0.3M HNO_3	Toxic, irritant, and oxidant
0.1M cadmium chloride in 0.3M HNO_3	Toxic and suspected carcinogen
0.1M copper(II) nitrate in 0.3M HNO_3	Toxic, irritant, and oxidant
0.1M Tin(IV) chloride in 0.3M HNO_3	Corrosive and irritant

- *Hazards of 6M ammonia, 6M hydrochloric acid, 6M nitric acid, 3M potassium hydroxide, and 1M thioacetamide are listed in the common reagents table in chapter 2.

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 II cations

1. Take 15 drops of the test solution if the group I cations are not present in the sample or take the **supernatant of step 1 of group I analysis**. Find its pH using a short-range pH paper. If the pH is 0.5 ±3 there is no need to adjust the pH. If pH is lower, increase it to 0.5 ±3 by adding drops of 0.5M ammonia solution, one drop at a time while stirring. If pH is higher, decrease to 0.5 ±3 by adding drops of 0.5M HCl, one drop at a time while stirring. Then add 10 drops of 1M thioacetamide stir and heat for 10 min in a water bath. Add 1 drop of 0.5M NH_3,stir, centrifuge for 2 min, and add 5 drops of 1M thioacetamide, stir, and heat again for 2 min. Cool in room temperature water bath and add 1 more drop of 0.5M ammonia while stirring and centrifuge for 2 min. Decant and **keep the supernatant** for group III cations and **keep the precipitate** for separation and analysis of group II cations. The precipitate may be one or more of the following: SnS_2 (yellow), CdS (yellow-orange), CuS (black-brown), Bi_2S_3 (black). Record the observation in the datasheet.
2. Wash the precipitate from step 1 by re-suspending it in 1 mL (20 drops) of 0.1M NH_4Cl, centrifuge for 2 min, decant, and discard the supernatant which is just the washing liquid. Re-suspend the precipitate in 1 mL (20 drops) of 3M KOH + 1 drop of 1M thioacetamide, stir, loosely stopper the test tube, and heat in a water bath for 2 min. Centrifuge the hot mixture for 2 min and decant while it is hot. **Keep the supernatant** for analysis of Sn^{4+} which exists as soluble $Sn(OH)_6^{2-}$ ion at this stage and **keep the precipitate**, if there is any, for analysis of the rest of the group II cations. Record the observation in the datasheet.
3. Add 6M HCl drop by drop to the supernatant from step 2 and keep testing with blue litmus paper until the mixture turns acidic. Then add 5 drops of 1M thioacetamide, stir, and heat in a water bath for 2 min. Yellow precipitate at this stage is SnS_2 which confirms Sn^{4+} is present in the test sample, no yellow precipitate means Sn^{4+} was not present. Record the observation in the datasheet and discard the mixture in a waste container.
4. Wash the precipitate from step 2 by re-suspending it in 10 drops of distilled water and then centrifuge for 2 min. Decant and discard the supernatant and wash the precipitate again by re-suspension in 10 drops of distilled water followed by centrifuge for 2 min, decant and discard the supernatant. Re-suspend the precipitate in 10 drops of distilled water + 2 drops of 6M HCl and heat for 2 min. Centrifuge and decant while the mixture is still hot. If the supernatant appears turbid due to some precipitate left in it, use the cotton plug technique to aspire clean supernatant and filter out the residual precipitate. **Keep the supernatant** for analysis of Cd^{2+} which may exist as dissolved $[CdCl_4]^{2-}$ ion at this stage and **keep the precipitate**, if there is any, for analysis of remaining group II cations. Record the observation in the datasheet.
5. Add 6M NH_3 drop by drop to the clear supernatant from step 4 and keep testing with red litmus paper until the solution turns basic. Add 2 drops of 1M thioacetamide, stir, and heat for 2 min in a water bath. If a yellow precipitate forms at this stage it is CdS that confirms Cd^{2+} was present in the test sample, otherwise Cd^{2+} was not present. Record the

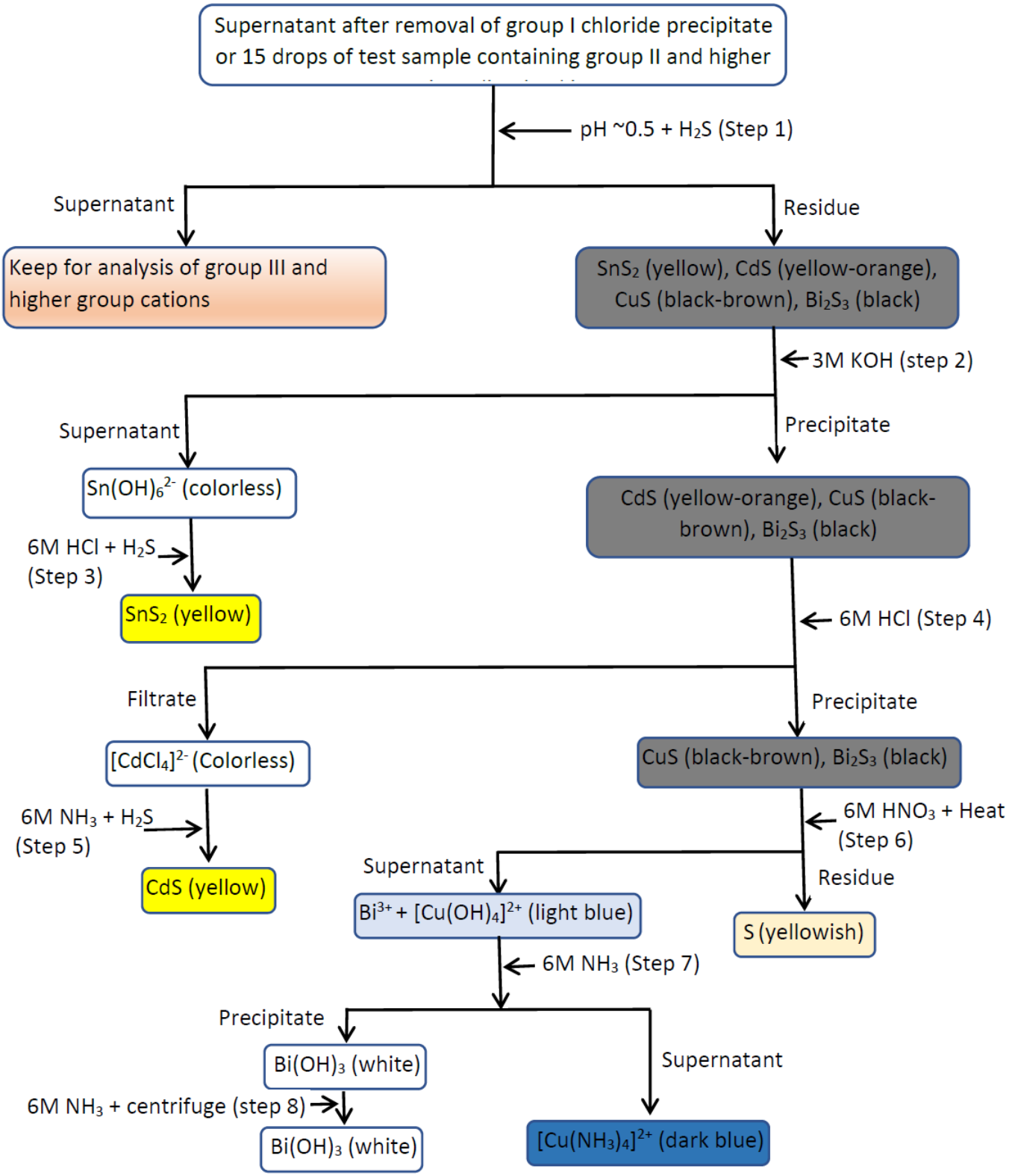
observation in the datasheet and discard the mixture in a metal waste container.

6. Wash the precipitate from step 4, if there is any, by re-suspending it in 10 drops of distilled water, centrifuge for 2 min, decant and discard the supernatant. Re-suspend the precipitate in 10 drops of 6M HNO_3 and heat in a boiling water bath for 5 min. The precipitate, i.e., CuS and/or Bi_2S_3 will dissolve in the liquid, and Cu^{2+} and/or Bi^{3+} hydrated ions and yellow sulfur particles may form. Remove the sulfur particles by centrifugation and decantation and discard them as there is no ion in them. **Keep the supernatant** for the analysis of Cu^{2+} and Bi^{3+} and record the observation in the datasheet.
7. Add 6M NH_3 drop by drop to the supernatant from step 6 and keep testing with red litmus paper till the solution turns alkaline. Add 10 more drops of 6M NH_3 solution after the solution turns alkaline to make it strongly alkaline. If the mixture becomes blue color at this stage, it is due to the blue $[Cu(NH_3)_4]^{2+}$ ion that confirms Cu^{2+} is present in the test solution. If there is a white suspension in the mixture, keep it for testing Bi^{3+}. Record the observation in the datasheet.
8. Centrifuge the mixture from step 7 for 2 min and decant and discard the supernatant. If there is any white precipitate left after decantation, it is most likely $Bi(OH)_3$. Wash the precipitate by re-suspending it in 10 drops of 6M NH_3, centrifuge for 2 min, and decant. If the white precipitate remains there after the washing, it is $Bi(OH)_3$ that confirms Bi^{3+} is present in the test solution, otherwise, Bi^{3+} is absent. Discard the mixture in a metal waste container and record the observation in the datasheet.

Datasheets filling instructions for group II 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-II Cations Analysis Flow Chart



Data sheet for known Group II cations (Sn^{4+}, Cd^{2+}, Cu^{2+}, and Bi^{3+}) 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 5		
Step 6		
Step 7		
Step 8		

Overall conclusion

Datasheet for unknown sample#_____ of Group II cations (Sn^{4+}, Cd^{2+}, Cu^{2+}, and Bi^{3+}) 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 5	
Step 6	
Step 7	
Step 8	

Overall conclusion