6.3: Procedure, flowchart, and datasheets for separation and confirmation of group IV and group V cations

Table 1. List of chemicals and their hazards*

Chemical	Hazard
6M Acetic acid (CH_3COOH)	Toxic and corrosive
0.2M Ammonium oxalate	Irritant
0.1M Barium chloride	Highly toxic
0.1M Calcium chloride	Irritant
0.1M Potassium chromate	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 IV and group V cations

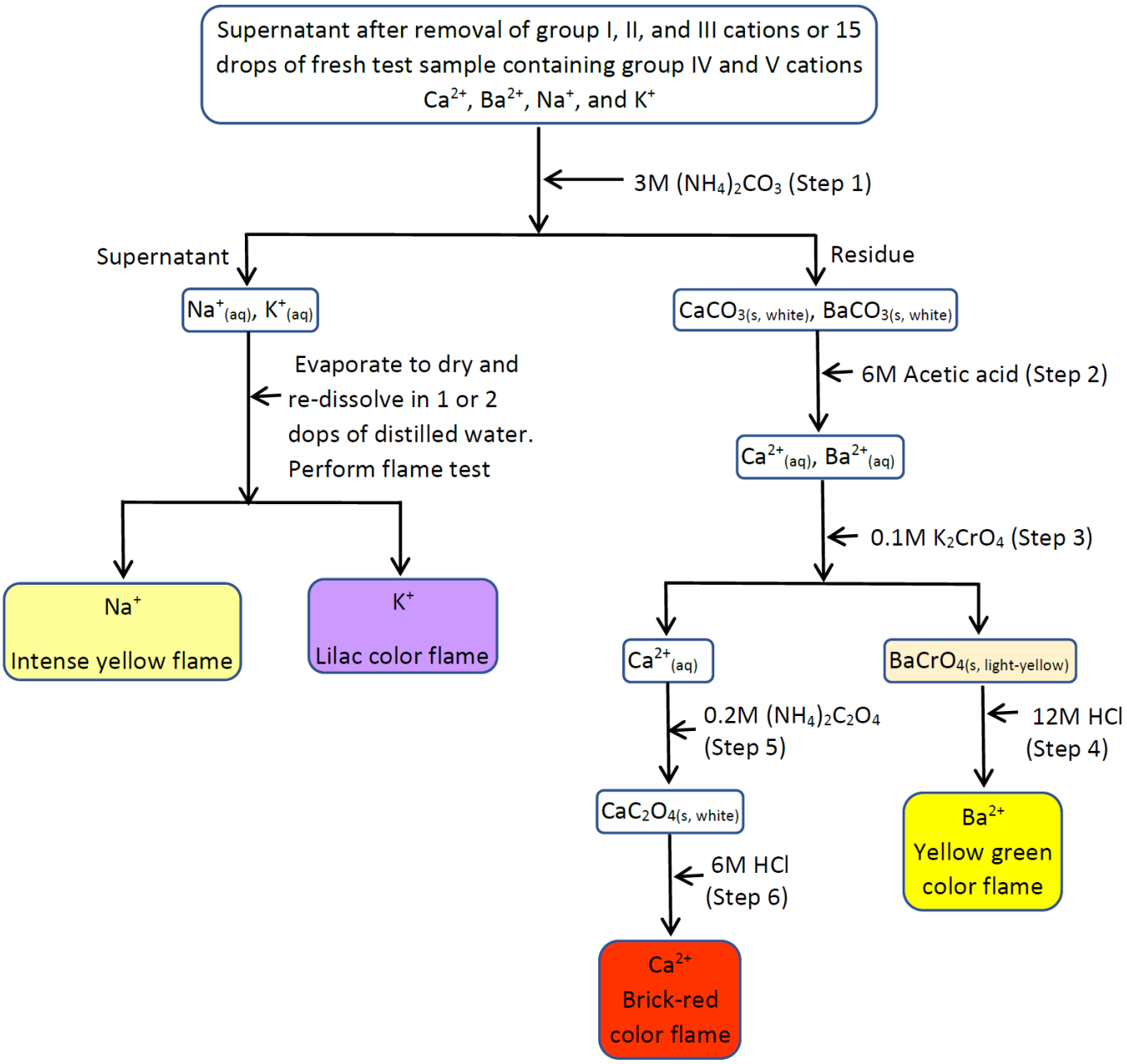
1. Take 15 drops of the fresh test solution if the group I to III cations are not present in the sample or **take the supernatant of step 2 of group III cations analysis**. Add 15 drops of 3M $(NH_4)_2CO_3$, stir to thoroughly mix using a clean glass rod, centrifuge for 2 min, decant and **keep the supernatant** for group V tests, and **keep the precipitate** for group IV cations analysis. Record the observations in the datasheet.
2. Wash the precipitate of step 1 by re-suspending it in 15 drops of distilled water under stirring, centrifuge for 2 min, decant and keep the precipitate and discard the supernatant which is just the washing liquid. Add 5 drops of 6M acetic acid to the precipitate and heat for half-min while stirring to dissolve the precipitate. Add 2 more drops of 6M acetic acid while heating and stirring if needed to fully dissolve the precipitate. After the precipitate has dissolved, add 3 more drops of 6M acetic acid to make a CH_3COOH/CH_3COO^- buffer. Record the observations in the datasheet.
3. Add 10 drops of 0.1M K_2CrO_4, stir to mix, and heat for 1 min. Immediately centrifuge for 2 min and decant while hot. **Keep the supernatant** for analysis of Ca^{2+}. If a light-yellow precipitate is formed at this stage it is most likely $BaCrO_4$ due to Ba^{2+} present in the test sample. **Keep the precipitate** for the flame test. Record the observations in the datasheet.
4. Wash the precipitate of step 2 by re-suspending it in 15 drops of distilled water, centrifuge for 2 min, decant, and discard the supernatant water which is just the washing solvent. Add 5 drops of 12M HCl to the precipitate, stir to mix, and heat in a boiling water bath for 2 min to dissolve the precipitate. Perform the flame test, i.e., dip a clean nichrome or platinum wire loop in the solution and then place the loop on the outer edge of a blue flame of a Bunsen burner, approximately halfway between the top and bottom of the flame and observe the flam color. If the solution imparts yellow-green color to the flame, it is due to barium ion confirming Ba^{2+} is present in the test sample. The nichrome wire can be re-used after dipping it in 6M HCl followed by making it red-hot in a flame. Repeat this process until the wire does not impart color to the flame. Then the wire can be re-used. Another approach is to cut off the end part of the wire that was dipped in the salt, make a new loop at the fresh end, and use it for the next flame test. Discard the solution in a metal waste container and record the observations in the datasheet.
5. To the supernatant from step 3, add 10 drops of 0.2M ammonium oxalate ($(NH_4)_2C_2O_4$), stir to mix, centrifuge for 2 min, decant, discard the supernatant and observe the precipitate. The formation of white precipitate at this stage is CaC_2O_4 which is a strong indication of Ca^{2+} is present in the test solution. **Keep the precipitate** for the flame test. Record the observation in the datasheet.

6. Dissolve the precipitate of step 5 in 3 drops of 6M HCl. Perform the flame test, i.e., dip a clean nichrome or platinum wire loop in the solution and then place the loop in the outer edge of a blue flame of a Bunsen burner, approximately halfway between the top and bottom of the flame and observe the flam color. If the solution imparts brick-red color to the flame, it is due to calcium ions confirming Ca^{2+} is present in the test sample. Discard the solution in the metal waste container and record the observations in the datasheet.
7. Group V cations: Evaporite excess water from the supernatant of step 1 by heating. If any solid residue is left it is due to group V cations, i.e., sodium, potassium, etc. Add a drop or two drops of water to dissolve the precipitate. Perform the flame test, i.e., dip a clean nichrome or platinum wire loop in the solution and then place the loop in the outer edge of a blue flame of a Bunsen burner, approximately halfway between the top and bottom of the flame and observe the flame color. If the solution imparts some color to the flame, it is due to group V cations: an intense yellow color flame confirms Na^{+} is present in the test solution, and purple or lilac color to the flame confirms K^{+} is present in the test solution. Discard the solution in the metal waster container and record your observations in the datasheet.

Datasheets filling instructions for group IV and group V 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-IV and Group V Cations Analysis Flow



Data sheet for known Group IV cations (Ca^{2+} and Ba^{2+}) and Group V cations (Na^{+} and K^{+}) 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	

Overall conclusion

Data sheet for unknown sample#_____ of Group IV cations (Ca^{2+} and Ba^{2+}) and Group V cations (Na^{+} and K^{+}) 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	
Overall conclusion	