Group IV and V Qualitative Analysis

Equipment

- 1-tray of dropper bottles
- 2-micro spatulas
- 2-wooden test tube blocks
- 4-medicine droppers
- 12-16 micro test tubes
- 2-micro test tube brushes
- 2-10 mL beakers
- 1-small graduated cylinder

Make labels for 6 dropper bottles and fill them with the following solutions:

- 6 M HCl
- 6 M NaOH
- 6 M HNO₃
- 6 M NH₄OH
- 6 M HC₂H₃O₂
- distilled water

Make 6-8 capillary droppers and 2-3 stirring rods.

Helpful tips

Centrifuge

In order to facilitate the separation of solid precipitate from supernatant liquid, a sample must be centrifuged. For the centrifuge to spin smoothly, it must be balanced. Be sure to place a like test tube with approximately the same amount of liquid just opposite your sample tube.

Complete Precipitation

When instructions require that a solution be made alkailne or acidic (in order to precipitate selected ions), be sure that you've added enough acid or base by swabbing a drop of the solution across a strip of litmus paper. Litmus turns blue when exposed to basic solutions and red when exposed to acidic solutions. Then THROW THE LITMUS PAPER AWAY.

Drop Size

It should be noted that the drops delivered by a capillary pipette are much smaller than a medicine dropper. When instructed to add a given number of drops of unknown or a necessary reagent, be sure to use a medicine dropper for big healthy drops.

Safety Precautions

Some unknowns contain solutions that may stain the skin. Treat them with care. The concentrated acids and bases will naturally cause skin irritations or burns, so if you should contact them, wash the solution off immediately.
Directions for Separation of Group IV Cations

To 5 drops of your Unknown solution (or a control sample) add 6 M NH₄OH until the solution is alkaline. Then add 5 drops of (NH₄)₂CO₃, stir, and place in a hot water bath for at least 2 minutes. Centrifuge.

Residue A
May contain BaCO₃, SrCO₃, and/or CaCO₃
Dissolve the residue in about 5 drops of 6 M HC₂H₃O₂. Heat in a water bath if necessary to get it to dissolve. Then add 2 drops of NH₄C₂H₃O₂ and 5 drops of K₂CrO₄. Mix well and place in a hot water bath for 1 minute. Centrifuge.

Filtrate A
Contains any Group V cations that may be present.
This can be discarded.

Residue B
May contain BaCrO₄ (yellow precipitate)
Add a few drops of 12 M HCl to the residue and make a flame test. Pale yellow-green flame confirms the presence of Ba²⁺ in your unknown.

Filtrate B
May contain Sr²⁺ and/or Ca²⁺
Add 8 drops of 6 M NH₄OH, 4 drops of K₂CrO₄, and 15 drops of alcohol. Mix well. Place in an ice bath and let stand 5-10 minutes. Centrifuge.

Residue C
May contain SrCrO₄
Add a few drops of 12 M HCl to the residue and make a flame test. A crimson flame confirms the presence of Sr²⁺ in your unknown.

Filtrate C
May contain Ca²⁺
Acidify with 6 M HC₂H₃O₂ and then add 10 drops of (NH₄)₂C₂O₄. A white precipitate indicates the presence of Ca²⁺ in your unknown.
Centrifuge and discard the liquid. Add a few drops of 12 M HCl to the residue and make a flame test. Orange-red flame confirms the presence of Ca²⁺ in your unknown.
Directions for Separation of Group V Cations

Take 5 drops of your test solution, add 5 drops of 12 M HCl and make a flame test.

A yellow flame that persists for at least 5 seconds confirms the presence of Na\(^{+1}\).
A pale violet flame that persists at least 2 seconds confirms the presence of K\(^{+1}\).

**NOTE:** (make the test with solutions containing only Na\(^{+1}\) or K\(^{+1}\) ions and then with a solution of mixed ions.) In the presence of the yellow sodium flame, the violet flame of the potassium will be hard to see with the naked eye. But if the flame is viewed through blue cobalt glass, the yellow sodium color will be absorbed by the blue glass and the violet potassium flame can be seen clearly.

**Test for NH\(_4\)\(^{+1}\)**

To 5 drops of your unknown solution, add 5 drops of 6 M NaOH. Warm gently in a hot water bath. If NH\(_4\)\(^{+1}\) ions are present, ammonia gas will be evolved and can be detected by its odor or by the blue color it gives to a piece of moistened red litmus paper held near the mouth of the tube.

**Test for Na\(^{+1}\)**

To 2 drops of your unknown solution, add 1 drop of 12 M HC\(_2\)H\(_3\)O\(_2\) and 2 drops of magnesium uranyl acetate reagent. Let stand for at least 5 minutes. A fine yellow crystalline precipitate that develops slowly confirms the presence of Na\(^{+1}\) in your unknown.

**Test for K\(^{+1}\)**

To 3 drops of your unknown, add 1 or 2 drops of sodium tetraphenyl boron, Na(C\(_6\)H\(_5\))\(_4\)B. If K\(^{+1}\) is present, a white precipitate will form immediately. Since NH\(_4\)\(^{+1}\) also gives a precipitate with this test, it is not confirmatory when NH\(_4\)\(^{+1}\) is present.

**Test for Mg\(^{+2}\)**

To 2 drops of your unknown solution, add 2 drops of NH\(_4\)Cl and enough 6 M NH\(_4\)OH to make the solution alkaline. (Mix the solution thoroughly with a stirring rod and test with litmus.) Add 3 drops of Na\(_2\)HPO\(_4\). A white crystalline precipitate confirms the presence of Mg\(^{+2}\) in your unknown.
REPORT SHEET  
Analysis of Unknown-Group IV

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<th>Reagent Added</th>
<th>Result</th>
<th>Conclusion</th>
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Ions Present in Unknown _________________________________
**REPORT SHEET**

Analysis of Unknown-Group V

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