

**Periodic Properties:
Analysis of Group I, II and Transition Metals**

Procedure

All solutions should be mixed thoroughly after any additions. Clearly record **all** observations (indicating what you did), even if a description of the the product is given in the procedure.

Part I

Identifying Group II Metals (Mg^{2+} , Ca^{2+} , and Ba^{2+})

1. Obtain approximately 1 mL of Group II known solution (about 20 drops) in a test tube.
2. Add 6 drops of 6 M NH_3 , followed by 4 drops of 6 M HCl . The acid-base reaction, which is exothermic, will produce fumes of NH_4Cl solid. Check that the solution is basic (a drop of the solution on a clean stirring rod turns indicator paper the proper color (on container)). If not, add 6 M NH_3 dropwise until just basic.
3. Add 1 M acetic acid dropwise to the solution until indicator paper shows the solution is slightly acidic to neutral (to indicator paper). Avoid using excess acetic acid.
4. Add 8 drops of 1 M K_2CrO_4 (yellow) to the solution. Be careful not to use $\text{K}_2\text{C}_2\text{O}_4$.
5. A yellow precipitate (BaCrO_4) should form. If it does not form, add more K_2CrO_4 . Centrifuge and decant the solution into a clean test tube. Save the solution for step 8 and use the precipitate in the next step.
6. Dissolve the precipitate in 3 drops of 6 M HCl and 3 drops of distilled water.
7. Add 20 drops of 0.1 M Na_2SO_4 . A white precipitate (BaSO_4) in a yellow solution will result. If you are not sure of the color of the precipitate, then centrifuge it, discard the solution and wash the precipitate with distilled water. This precipitate confirms the presence of Ba^{2+} .
8. Add 3 drops of 1 M $\text{K}_2\text{C}_2\text{O}_4$ to the solution from step 5. A white precipitate (CaC_2O_4) should form. It may happen slowly.
9. Centrifuge and decant the solution into a clean test tube and save for step 11 and use the precipitate in the following step.
10. Dissolve the precipitate in 4 drops of 6 M HCl ; then add 20 drops of distilled water. Add one drop of 1 M $\text{K}_2\text{C}_2\text{O}_4$ and make the solution basic (to indicator paper) with 6 M NaOH . A white precipitate (CaC_2O_4) confirms the presence of Ca^{2+} .

11. Add 6 drops of 6 M NaOH to the solution from step 9. Centrifuge. A translucent, pale white to yellow precipitate ($\text{Mg}(\text{OH})_2$) should now be visible. Withdraw the solution from the top with a dropper and discard. Use the precipitate in the next step.
12. Dissolve the precipitate in 3 drops of 6 M HCl.
13. Add drops of 6 M NH_3 until a drop of the solution turns indicator paper basic.
14. Add 10 drops of 1 M Na_2HPO_4 . A white precipitate (MgHPO_4), which may form slowly, confirms the presence of Mg^{2+} .

Identifying Transition Metals (Fe^{3+} , Cu^{2+} , and Cr^{3+})

1. Obtain approximately 1 mL of the Transition Metal known mixture in a test tube.
2. Add 20 drops of 6 M NH_3 .
3. A mixture of precipitates ($\text{Fe}(\text{OH})_3$ and $\text{Cr}(\text{OH})_3$) will form. Centrifuge and decant the solution into a clean test tube. Save this solution (dark blue, $\text{Cu}(\text{NH}_3)_4^{2+}$) for step 10 and use the precipitate in the following step.
4. Wash the precipitate with about 1 mL of distilled water from your wash bottle. Centrifuge. Discard the water and use the precipitate in the next step.
5. Add 12 drops of 3% H_2O_2 and 5 drops of 6 M NaOH to the precipitate. Let this mixture stand for about 1 minute.
6. Centrifuge this mixture. Save the solution (it should now be yellow, indicating CrO_4^{2-} ion produced from the hydrogen peroxide oxidation of $\text{Cr}(\text{OH})_3$) in a clean test tube for step 11. Use the precipitate ($\text{Fe}(\text{OH})_3$) in the following step.
7. Wash the precipitate in the same manner as in step 4.
8. Dissolve the precipitate with 5 drops of 6 M HNO_3 . Add 3 mL of distilled water.
9. Add 1 drop of 0.1 M KSCN to this solution and mix thoroughly. A deep red color (a **solution** of FeSCN^{2+}) confirms the presence of Fe^{3+} .
10. Make the solution from step 3 acidic by adding drops of 6 M acetic acid until the solution turns distinctly **light** blue (Cu^{2+}). Add 10 drops of 0.1 M $\text{K}_4\text{Fe}(\text{CN})_6$ and centrifuge. A red-maroon precipitate ($\text{Cu}_2\text{Fe}(\text{CN})_6$) confirms the presence of Cu^{2+} .
11. Make the solution from step 6 acidic by adding drops of 6 M acetic acid until the solution turns indicator paper acidic to neutral. Add 5 drops of 0.1 M $\text{Pb}(\text{NO}_3)_2$. Centrifuge. A yellow precipitate (PbCrO_4) confirms the presence of Cr^{3+} .

Identifying Group I Metals (Li^+ , Na^+ , and K^+)

1. Obtain separate samples of 1.0 M K^+ , 1.0 M Li^+ , and 1.0 M Na^+ (as the chlorides).
2. Light a Bunsen burner and adjust the air to give a **stiff blue flame**.
3. Dip the loop of your wire into 6 M HNO_3 and hold it at the top of the **inner** blue cone of the Bunsen burner flame until the wire glows and gives off almost no flame.
4. After the wire has been cleaned, dip the loop in the 1.0 M K^+ solution and hold it in the flame. Observe the first color produced in the flame.
5. Repeat steps 3 and 4 with 1.0 M Li^+ , and 1.0 M Na^+ .

To help you follow the chemistry, page 5 contains flow charts of the Group II and Transition Metals procedures. A vertical double line indicates precipitates and the step number (of the procedure) is given in parentheses. Note the various separation and identification steps.

Part II

You will obtain **three** unknown solutions. Each unknown contains **one ion** (Group I) **or one, two or all three ions** (Group II and Transition Metals). Using the procedures and observations you made for the known mixtures, you will identify the ion in each unknown. Record the number of your unknown solutions and follow the Part I procedures (use the flow chart as a guide) on 10 drop samples of the unknowns. Remember, not all procedures will give identical observations with what you saw in part I because the unknown may not contain all the ions.

Questions

1. **Refer to the flowchart** on the page 5 to answer the following:
 - a. From your knowledge of solubility rules, what does a double line mean?
 - b. What does a branch in the chart mean?
 - c. Is magnesium chromate (MgCrO_4) soluble or insoluble in a mixture of NH_3 and acetic acid solution? How do you know?
 - d. Which ion's confirmation test (from Group II and the Transition Metals) does **not** rely on the observation of a **precipitate**?

Data Treatment and Discussion

Your careful and detailed observations, in your laboratory notebook, are your "data" for this experiment. Attempt to be thorough. Indicate which cations were present in your unknowns along with a **discussion** of what, **specifically**, allowed you to make these conclusions.

Conclusion

Give the **unknown numbers** and the **ions** you identified. Also, address the following:

If you wanted to include Sr^{2+} analysis in the Group II ions, what **specific** tests (indicate the test and any ions needed) and possible observations would be needed before it could be included? You do not need to know the results of the tests.

Flow Charts of the Separation and Confirmation of Group II and Transition Metals

