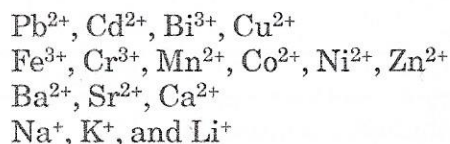


INTRODUCTION TO THE CATIONS

The following analytical procedure will lead to the correct evaluation of an unknown if the following ions only are present:



If other ions are present (only the ones listed above will possibly be present in your unknown), the procedure must be modified.

The known solution is already prepared for you. Analyze the known solution (as well as the unknown solution) following the Procedure as described on the following pages. Analysis of the known will show you what results to observe for a positive test.

Obtain an unknown from your instructor (during the first working period on the cation analysis). This unknown contains any or all the ions listed in the first paragraph above. Note that while you are doing the known solution, you may ask any question concerning the chemistry, colors, amount of precipitate, what different terms mean, etc. However, during the unknown the instructor will not help you in deciding whether the particular ion is positive or not. For example, he will not answer questions as "Is this precipitate red enough to be a positive test?" or "Is there enough precipitate to be considered a positive test?" You should have deduced what is red enough or the amounts necessary for a positive test from the known solution.

After you have completed the testing of all 16 cations, you will submit on a report (found at the end of this chapter) the cations (written in ionic form, e.g., Co^{2+} , Cr^{3+} , Zn^{2+} , etc.) that you have found to be present in your known. The report must be written in ink.

Since the unknown investigated in this experiment contains fewer ions than are included in most test procedures, a modified analytical scheme is presented below. An outline (or schematic) of the chemistry involved in the qualitative scheme is included in this chapter. Remember that the entire analysis can be done with 3 mL of the unknown solution. If the volumes become excessively large (above 7 to 8 mL), evaporate the solution (using the evaporating dish in the hood) to 3 mL.

You should read (review) the qualitative techniques presented in Appendix IV to ensure a greater chance of doing a successful analysis.

Traditionally, cation analysis starts with the analysis of Ag^+ and Hg_2^{2+} . However, this group has been eliminated from our analysis. Hence, Group II is our beginning point in the Cation Analysis.

PROCEDURE

In this method of analysis the identification of the cations will be done in four parts or groups. The four procedural groups that are to be used for the analysis of your cation unknown are given on the next several pages.

GROUP II
TESTING FOR: Pb^{2+} , Bi^{3+} , Cu^{2+} , Cd^{2+}

Take 3 mL of your original unknown solution to begin your analysis with Step A of Group II.

<i>Step</i>	<i>Purpose</i>	<i>Procedure</i>	<i>Results</i>
A	Adjusting pH and pptn of Group II sulfides	Test the original 3 mL of unknown solution with methyl violet paper (MVP) (NOTE 2). If MVP turns yellow, then add 6 M NH_4OH dropwise with mixing until the first drop causes the MVP to become a dark green or blue-green color. If MVP stays violet, add 6 M HCl dropwise until the first drop causes MVP to become dark green or blue-green in color (NOTE 3). Soln should be clear once the pH is properly adjusted. Add 15 drops of thioacetamide, mix well, and heat in hot water bath for 10 minutes (NOTE 4). Then centrifuge, decant the liquid (Groups III–V), and save the ppt for Group II. Recheck the pH of the liquid with MVP (adding 6 M HCl or NH_4OH if MVP is not dark green). To this liquid add 15 more drops of thioacetamide, mix well, and heat it for 10 minutes to be sure that all of Group II is removed from the liquid. If more ppt forms, centrifuge, decant this liquid (which is to be saved for Groups III–IV, and wash this ppt into the first one (the test tube containing the ppt for Group II). Centrifuge the test tube containing Group II and the water, save the ppt, and throw away the washings. Wash the ppt again. Save the ppt for Step B and throw away the washings.	<u>PbS</u> (black) <u>Bi_2S_3</u> (black or brown) <u>CuS</u> (black) <u>CdS</u> (brownish-orange) or (yellow)
B	Dissolving PbS , Bi_2S_3 , CuS , CdS	To the ppt from Step A, add 2 mL of 6 M HNO_3 . Heat with stirring for 5 minutes. Centrifuge, save the liquid for Step C and discard the ppt (if any is present) (NOTE 5).	Pb^{2+} , Bi^{3+} , and Cd^{2+} are colorless ions and Cu^{2+} is light blue
C	Sep. of $PbSO_4$	To the liquid from Step B, add 1 mL of distilled water and 2 grams (amount equal to the size of two lima beans) of solid $(NH_4)_2SO_4$. Shake well. Centrifuge and decant the liquid, which may contain Bi^{3+} , Cu^{2+} , and Cd^{2+} (Step E). Use the ppt in Step D.	<u>$PbSO_4$</u> (white)

Step	Purpose	Procedure	Results
D	Ident. of Pb^{2+}	Dissolve the white ppt from Step C in 20 to 25 drops of 3 M $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ (ammonium acetate). Warm (NOTE 6). Add 10 drops of 6 M $\text{HC}_2\text{H}_3\text{O}_2$ and 5 drops of 1 M K_2CrO_4 . Mix well. Centrifuge. Discard the liquid and to the yellow ppt, add 3 mL of 6 M NaOH. Mix well. If at least 90% of the yellow ppt dissolves, then Pb^{2+} was present in the unknown.	PbCrO_4 (yellow)
E	Sep. and Ident. of Bi^{3+}	To the liquid from Step C add 2 mL of conc NH_4OH slowly and carefully. Mix well. Centrifuge the mixture. Decant the liquid and save it for Steps F and G. Wash the ppt with two 2 mL portions of distilled water (discard the washings). To the ppt add 2 mL of freshly prepared Na_2SnO_2 soln (Prepare the Na_2SnO_2 soln by using the instructions in NOTE 7). Blackening of the ppt proves Bi^{3+} .	$\text{Bi}(\text{OH})_3$ white ↓ Bi^0 black
F	Ident. of Cu^{2+}	If the liquid from Step E is dark blue, Cu^{2+} is present. <i>Only if the soln color is doubtful, make 1/2 of the soln from Step E acidic with 6 M $\text{HC}_2\text{H}_3\text{O}_2$ and add a few drops of $\text{K}_4\text{Fe}(\text{CN})_6$ (potassium ferrocyanate). (Save the other 1/2 of the soln for step G). A red, brownish-red, or maroon ppt proves Cu^{2+} (NOTE 8).</i>	$[\text{Cu}(\text{NH}_3)_4]^{2+}$ (dark blue) $\text{Cu}_2\text{Fe}(\text{CN})_6$ (maroon)
G	Ident. of Cd^{2+}	To the other half of the soln saved from Step E add 0.9 g of $\text{Na}_2\text{S}_2\text{O}_4$ (sodium dithionite), mix well, and heat the solution for 10–15 minutes in the hot water bath. Centrifuge the mixture, decant the liquid (save it), and discard the ppt (if present). (NOTE 9) If the soln is still blue at this point, see your instructor. To the liquid add 10 drops of 5% thioacetamide and heat for 5 to 8 minutes in a hot water bath. The formation of a brownish-orange or yellow ppt indicates the presence of Cd^{2+} .	CdS (brownish-orange) or (yellow)

GROUP II NOTES

1. Only about 3 mL of unknown are needed to do the entire cation analysis; though more than that is given to you in case you need to repeat some tests.
2. At this pH only the sulfides of Group II will ppt. Groups III, IV, and V are in soln. Methyl violet paper is green or blue-green at pH = 0.5, violet if too basic, and yellow if too acidic.

3. BiOCl (white) and the hydroxides of Fe³⁺ (reddish-brown), Cr³⁺ (gray-green), and Mn²⁺ (pink) will ppt. on addition of NH₃ (ammonium hydroxide). The four hydroxides dissolve on addition of acid. If a white ppt. is still present after the pH has been adjusted to 0.5, it may be PbCl₂ or BiOCl (or both). Continue with the thioacetamide treatment, for these respective chloride salts will be converted to their respective sulfide salts.
4. Thioacetamide hydrolyzes as follows when heated in acidic soln.:

$$\text{CH}_3\text{CSNH}_2 + \text{H}^+ + 2 \text{H}_2\text{O} \longrightarrow \text{CH}_3\text{COOH} + \text{NH}_4^+ + \text{H}_2\text{S}$$
5. The sulfides dissolve in HNO₃ according to the equation:

$$3 \text{PbS} + 8 \text{HNO}_3 \longrightarrow 3 \text{Pb}(\text{NO}_3)_2 + 3 \text{S}^0 + 2 \text{NO} + 4 \text{H}_2\text{O}$$

If a white or yellow ppt. remains (or floats), it is only elemental sulfur (resulting from the oxidation of S²⁻).
6. Pb(C₂H₃O₂)₂, a soluble but slightly ionized salt, is formed.
7. Na₂SnO₂ is prepared as follows: to 1 mL of 0.1 M SnCl₂ (stannous chloride) soln. add 6 M NaOH dropwise with stirring until a permanent ppt. is formed. Continue the addition until the ppt. redissolves. The reaction of sodium stannite with bismuth (III) hydroxide is:

$$3 \text{SnO}_2^{2-} + 2 \text{Bi}(\text{OH})_3 \longrightarrow 3 \text{SnO}_3^{2-} + 2 \text{Bi}^0 + 3 \text{H}_2\text{O}$$
8. If Cd⁺ is present, Cd₂Fe(CN)₆ will ppt. Since Cd₂Fe(CN)₆ is only pale yellow in color, it will not interfere with the maroon color of the Cu₂Fe(CN)₆ ppt.
9. Na₂S₂O₄, sodium dithionite, is a strong reducing agent. It reduces Cu²⁺ to Cu⁰, thus removing Cu²⁺ from the soln. However, Cd²⁺ is not reduced. When thioacetamide is added to produce H₂S, only CdS (brownish-orange or yellow ppt) forms, not the black CuS ppt, which would have masked the CdS ppt.

GROUP III

TESTING FOR: Co²⁺, Ni²⁺, Mn²⁺, Fe³⁺, Cr³⁺, Zn²⁺

Step	Purpose	Procedure	Results
A	Pptn of Group III	To the liquid from Group II (NOTE 1) add 15 drops of conc NH ₄ OH and mix well. If the soln is not basic to litmus, add another 5 drops of conc NH ₄ OH and mix well. (Repeat with another 5 drops if the soln is not basic to litmus.) Now add 15 drops of 5% thioacetamide to this liquid. Place this mixture in a hot water bath for 12 minutes (NOTE 2, 3). Centrifuge and decant the liquid into a test tube labeled Group IV (to be used later). Wash the ppt with 2 mL of distilled water. Discard the wash water.	<u>CoS</u> <u>NiS</u> black black <u>MnS</u> <u>ZnS</u> pink white <u>FeS</u> <u>Cr(OH)₃</u> black grey-green

<i>Step</i>	<i>Purpose</i>	<i>Procedure</i>	<i>Results</i>
B	Sep. of Co and Ni from the other sulfides and hydroxide	To the ppt from Step A add 3 mL of 0.5 M HCl. Note, any acid or base soln that is not dilute (6 M) or concentrated (see page 169 for concentrations), must be prepared from the dilute (6 M) acid or base. If you have forgotten how to do dilutions, see technique 7 in Appendix IV (pg 298–9). Mix well and separate immediately (NOTE 4). Save the liquid for Step F. To the ppt add 10 drops of 0.5 M HCl, shake well, and then add 1 mL of distilled water. Mix well, centrifuge, and separate immediately. Add this soln also to the liquid for Step F and save the ppt for Step C.	<u>CoS</u> <u>NiS</u> black black (remains) Mn ²⁺ , Zn ²⁺ , Cr ³⁺ , Fe ²⁺ (dissolve yielding these ions in soln)
C	Dissolving of CoS and NiS	To the ppt from Step B add 3 drops of conc HCl and 1 drop of conc HNO ₃ . Heat the mixture in a hot water bath for 6 minutes (NOTE 5). Dilute the solution with 3 mL of distilled water. Mix well. Remove any sulfur (NOTE 6) that forms (by centrifuging and decanting or by picking out the yellow or white particles with a glass rod). Make the soln just basic to litmus with 6M NH ₄ OH (NOTE 7) by adding 3 drops of 6M NH ₄ OH at a time, mixing with a glass stirring rod, and touching the red litmus paper to see if it turns blue. Repeat the addition of 6M NH ₄ OH until the solution is basic (red litmus turning blue). Then add 3 extra drops of the 6M NH ₄ OH.	Co ²⁺ and Ni ²⁺ in soln
D	Ident. of Ni ²⁺	To 1 mL of soln from Step C, add 10 drops of dimethylglyoxime soln. The appearance of a pink or red ppt proves Ni ²⁺ (NOTE 8).	<u>Ni(DMG)₂</u> red
E	Ident. of Co ²⁺	To another 1 mL portion of the soln from Step C add 6 M HCl until the soln is acid to litmus. Add around 0.4 g of KSCN to this soln. Next add 25 drops of isoamyl alcohol-ether soln. Mix well. If Co ²⁺ is present, a blue or blue-green color will appear in the isoamyl alcohol-ether layer (NOTE 9). If the isoamyl alcohol-ether layer is red (NOTE 9), add around 0.3 g of solid NH ₄ HF ₂ (ammonium bifluoride).	Co(SCN) ₄ ²⁻ blue or blue-green soln in alcohol- ether layer

Step	Purpose	Procedure	Results
		If the color remains, add more NH_4HF_2 , mix well, and observe the color in the isoamyl alcohol-ether layer. (Do not get any of the solid NH_4HF_2 , or the soln containing it, on your skin or fingernails; if you do, wash it off immediately with water.)	
F	Sep. of Fe^{3+} and Mn^{2+} from Zn^{2+} and Cr^{3+}	(Note: If the liquid saved for Step F is not clear, add solid NaCl , about the size of a green pea, mix well, and centrifuge. Throw away the ppt and save the solution; if the solution is still cloudy, filter the solution with filter paper, throw away the ppt, and continue with the filtered soln in Step F.) There should be 4 mL or less of soln. (If there are more than 4 mL, evaporate the soln in an evaporating dish in the hood until there are only 3 mL left.) To this soln add 6 M NaOH until the soln is basic to litmus. Then add 15 more drops of 6 M NaOH . Next, slowly add 0.4 g of Na_2O_2 (sodium peroxide), to the mixture (the initial reaction may occur with rapid evolution of oxygen and cause the solution to foam out of the test tube. Therefore, add the Na_2O_2 (any excess solid Na_2O_2 not used is to be discarded in the sink with water flushing—do not put into the trash can) slowly and warm the soln gently for the first minute of the 10-minute heating period) and heat in a hot water bath for 10 minutes (or longer if oxygen gas bubbles from the Na_2O_2 decomposition is still occurring; then continue heating until oxygen gas bubbles cease to form—NOTE 10). Centrifuge. Save the liquid for Steps J and K. Add one mL of 0.5 M NaOH to the ppt, mix well, and centrifuge. Add this liquid to that saved for Step J and K, and save the ppt for Step G.	$\frac{\text{Mn(OH)}_3}{\text{brown}}$ $\frac{\text{MnO}_2}{\text{black}}$ and $\frac{\text{Zn(OH)}_4^{2-}}{\text{colorless}}$ $\frac{\text{Fe(OH)}_3}{\text{red-brown}}$ ppt CrO_4^{2-} (yellow soln)
G	Dissolving of Mn(OH)_3 and Fe(OH)_3	Treat the ppt from Step F with 2 mL of 6 M HNO_3 and 4 drops of 1 M NaNO_2 (sodium nitrite) (NOTE 11). Stir the mixture and heat in a hot water bath for 10 minutes. Centrifuge the mixture. After decanting the solution (throw away any ppt that remains here); cool the soln (to be used for Steps H and I).	Mn^{2+} and Fe^{3+} pale reddish pink brown <u> </u> solution

<i>Step</i>	<i>Purpose</i>	<i>Procedure</i>	<i>Results</i>
H	Ident. of Fe ³⁺	To 1 mL of the soln from Step G, add 5 to 8 drops of 1.0 M KSCN (potassium thiocyanate). A deep blood-red soln proves Fe ³⁺ (NOTE 12).	FeSCN ²⁺ dark (blood) red soln
I	Ident. of Mn ²⁺	To another 1 mL portion of the soln from Step G, add 5 drops of 6 M HNO ₃ and 0.2 g (about the size of a green pea) of solid NaBiO ₃ . The formation of a dark pink, red-purple, or purple colored soln (centrifuge, if necessary) confirms Mn ²⁺ . If the dark pink, red-purple, or purple color forms and then disappears, add another 0.2 g amount of NaBiO ₃ (sodium bismuthate) (NOTE 13).	MnO ₄ ⁻ purple soln
J	Ident. of Cr ³⁺	To 1/3 of the liquid from Step F, add 6 M HC ₂ H ₃ O ₂ until the soln is acidic to litmus. Then add 4 drops of 0.1 M Pb(C ₂ H ₃ O ₂) ₂ . The formation of a yellow ppt proves Cr ³⁺ .	<u>PbCrO₄</u> yellow
K	Ident. of Zn ²⁺	To another one-third of the liquid from Step F, add 6 M HCl until soln is acidic to litmus (mix well). Then add 7 more drops of 6 M HCl and 4 drops of 4 M ethylenediamine soln (NOTE 14). Mix well. To this soln add 10 drops of K ₄ Fe(CN) ₆ (potassium ferrocyanate) soln. A white or beige colored ppt (NOTE 15), Zn ₂ Fe(CN) ₆ , proves the presence of Zn ²⁺ (NOTE 16).	<u>Zn₂Fe(CN)₆</u> white

GROUP III NOTES

1. If a ppt. is present in the liquid saved for Group III, centrifuge and use this liquid for Group III. To the ppt. add 0.5 mL conc. HNO₃ and stir. (If the ppt. does not dissolve, add one drop of conc. HCl). Then add 2 mL of distilled water. To 1/2 of the soln do the Mn²⁺ test (step I) and to the other 1/2 do the Fe³⁺ test (step H).
2. Under basic H₂S conditions the equilibrium (overall), $\text{H}_2\text{S} \rightleftharpoons 2\text{H}^+ + \text{S}^{2-}$, is shifted to the right; thus causing a high concentration of S²⁻ and allowing those sulfide ppts. (Group III) that are not as insoluble (as those of Group II are) to be precipitated. Cr(OH)₃ is more insoluble than Cr₂S₃.
3. Since S²⁻ is a reasonable reducing agent, Fe³⁺ is reduced to Fe²⁺, yielding the FeS ppt.

4. All the sulfides or hydroxides are soluble in very dilute acid solutions (as expected; see NOTE 2) except CoS and NiS. When CoS and NiS precipitate, they change from the α to the β crystalline form. The β form is more insoluble but it still dissolves at a slow rate; hence the necessity to do the separation quickly.
5. Heating (also removes the oxides of nitrogen, which may interfere with the Ni^{2+} and Co^{2+} tests) causes the sulfides to dissolve as shown in the following reactions:

$$3 \text{CoS} + 8 \text{H}^+ + 2 \text{NO}_3^- \longrightarrow 3 \text{Co}^{2+} + 2 \text{NO} \uparrow + 3 \text{S}^0 \downarrow + 4 \text{H}_2\text{O}$$

$$3 \text{NiS} + 8 \text{H}^+ + 2 \text{NO}_3^- \longrightarrow 3 \text{Ni}^{2+} + 2 \text{NO} \uparrow + 3 \text{S}^0 \downarrow + 4 \text{H}_2\text{O}$$
6. HNO_3 is a good oxidizing agent and it oxidizes S^{2-} to S^0 . S^0 is a yellow or white ppt.
7. Hydrated Ni^{2+} and Co^{2+} are converted to $[\text{Ni}(\text{NH}_3)_6]^{2+}$ (blue) and $[\text{Co}(\text{NH}_3)_6]^{2+}$ (pink) ions.
8. Centrifuge and observe the color of the soln. If the soln has turned brown upon the addition of dimethylglyoxime, then Co^{2+} may be present. $\text{Co}(\text{DMG})_2$ is a brown water soluble complex.
9. Addition of KSCN converts $\text{Co}(\text{NH}_3)_6^{2+}$ to $[\text{Co}(\text{SCN})_4]^{2-}$ (blue in the alcohol-ether layer) and $[\text{Fe}(\text{NH}_3)_6]^{3+}$ (contaminant, which should not be there, present even in trace amounts causes the following problem) is converted to FeSCN^{2+} , which is red or purple in the alcohol-ether layer. The addition of NH_4HF_2 causes the FeSCN^{2+} to be converted to the colorless FeF_6^{3-} ion while the $[\text{Co}(\text{SCN})_4]^{2-}$ ion remains as is.
10. The HNO_3 (good oxidizing agent) treatment converts Fe^{2+} to Fe^{3+} and the Na_2O_2 (good oxidizing agent) along with NaOH converts $\text{Mn}(\text{OH})_2$ (white to pink ppt.) to $\text{Mn}(\text{OH})_3$ (brown ppt.) and MnO_2 (black ppt.). Cr^{3+} is oxidized to CrO_4^{2-} .
11. The NaNO_2 (mild reducing agent) causes Mn^{3+} and Mn^{4+} to be reduced to Mn^{2+} .
12. This is a very sensitive test for Fe^{3+} . Many chemicals (plus those introduced by sloppy lab procedures) contain a trace amount of Fe^{3+} that cause a pale red color to be observed. This is a negative test for Fe^{3+} . If the blood-red color forms (stays for at least 2 minutes) and then disappears, Fe^{3+} is still present.
13. NaBiO_3 (good oxidizing agent) will oxidize Mn^{2+} to MnO_4^- . However, if other oxidizing substances, which should have been removed, are still present, the purple color may appear and then disappear. Hence, the added amount of NaBiO_3 must be added to oxidize these species and the Mn^{2+} . If you lost a large amount of your Mn^{2+} , then you may only see a pink rather than a purple color. If the Mn^{2+} concentration is large, then the original solution will be pink and you should definitely see a purple color form in this confirmation test.

14. If a ppt. forms after the addition of ethylenediamine, then add 6 N HCl drop by drop until the ppt. redissolves.
15. $K_4Fe(CN)_6$ will give gray-green ppts. with Co^{2+} , Ni^{2+} , and Cr^{3+} , which should not be present or precipitate at this point in the scheme.
16. Alternate Zn^{2+} test: To the other 1/3 of the liquid saved from step F, add 10 drops of 5% thioacetamide and heat for 5 minutes in a hot water bath (in hoods). The formation of a white or gray ppt. that is soluble in HCl (centrifuge the solid down, remove the liquid, add one mL of 6 M HCl directly on the solid, and mix well) proves that Zn^{2+} is present. A white solid, free sulfur, may form by the CrO_4^{2-} oxidation of S^{2-} . However, sulfur will not be soluble in HCl.

GROUP IV
TESTING FOR: Ba^{2+} , Sr^{2+} , Ca^{2+}

Step	Purpose	Procedure	Results
A	Pptn of Group IV ions	Use all the liquid saved from Group III. However, if there are more than 4 mL, evaporate to 3 mL in an evaporating dish and transfer it to a clean test tube (if a black ppt is present, centrifuge, throw away the black ppt and continue with the liquid). Check the liquid to be sure it is alkaline to red litmus paper. If the liquid is not alkaline, add 6 M NH_4OH (while mixing well) to make the soln alkaline. Then add 7 drops of 6 M NH_4OH in excess. Add 1 M $(NH_4)_2CO_3$ until complete precipitation (if no ppt forms, add 25 drops of 1 M $(NH_4)_2CO_3$ and leave the test tube in an ice bath for 5 minutes) has been achieved (NOTE 1). Centrifuge and decant the liquid. Save the ppt for Step B and discard the liquid.	<u>$BaCO_3$</u> white <u>$SrCO_3$</u> white <u>$CaCO_3$</u> white
B	Sep. and Ident. of Ba^{2+}	Wash the ppt with 2 mL of distilled water. Throw away the washings. Add to the ppt a mixture of 12 drops of 6 M $HC_2H_3O_2$ and 24 drops of 3 M $NH_4C_2H_3O_2$. Mix well. To the resulting mixture add 1 M K_2CrO_4 drop by drop until complete precipitation has occurred. The formation of a yellow ppt indicates the presence of Ba^{2+} (NOTE 2). Centrifuge, decant, and save the liquid for Step C. Dissolve the yellow ppt in one mL of conc. HCl (heat soln for 5 minutes in a hot water bath with	<u>$BaCrO_4$</u> yellow

<i>Step</i>	<i>Purpose</i>	<i>Procedure</i>	<i>Results</i>
		occasional stirring; cool slightly before adding the 3 M H ₂ SO ₄) (NOTE 3). To 1/2 of the soln add 4 drops of 3 M H ₂ SO ₄ . A white ppt proves the presence of Ba ²⁺ . A flame test (NOTE 4) could be done on the other 1/2 of the soln. However, this is usually not necessary.	
C	Sep. and Ident. of Sr ²⁺	To the liquid from Step B add 6 M NH ₄ OH until the color changes from orange or gold to yellow. Then add 5 more drops of 6 M NH ₄ OH and 5 drops of 1 M K ₂ CrO ₄ . Add a volume of ethyl alcohol equal to the volume of the soln and mix very well. Cool the test tube for 3 minutes in an ice bath. The formation of a fine yellow ppt proves the presence of Sr ²⁺ (NOTE 5). Centrifuge and save the liquid for Step D. A flame test (NOTE 6) could be done on the yellow ppt. However, this is usually not necessary.	<u>SrCrO₄</u> yellow
D	Ident. of Ca ²⁺	Heat the liquid gently (NOTE 7) from Step C for 10 minutes in a hot water bath. Then add 4 drops of 0.4 M (NH ₄) ₂ C ₂ O ₄ , mix well, and cool for 5 min in an ice bath (NOTE 8). The formation of a white ppt proves the presence of Ca ²⁺ . A flame test (NOTE 9) could be done on the white ppt. However, this is usually not necessary. However, if you have any doubts about the ions present in Group IV, then it is highly recommended that you do the flame test as described in NOTE 10.	<u>CaC₂O₄</u> white

GROUP V
TESTING FOR Li^+ , K^+ , Na^+

<i>Step</i>	<i>Purpose</i>	<i>Procedure</i>	<i>Results</i>
A	Flame test for each ion	<p>To test for Group V ions (Na^+, K^+, and Li^+) do a flame test on the original unknown solution. Na^+ will give a persistent [<i>The Na^+ and Li^+ give persistent flame tests (they last over one minute), while K^+, Ba^{2+}, Sr^{2+}, and Ca^{2+} flame tests are not persistent (they last only 2–10 seconds)] yellow-orange flame; K^+ will give a light purple flame; and Li^+ will give a persistent bright red flame. If Na^+ is present, its yellow-orange flame will mask the light purple flame of K^+ and the bright red flame of Li^+. Therefore, if Na^+ is present, look at the flame through cobalt glasses. The cobalt glass in the lenses will absorb (hence, remove) the yellow-orange light from the Na^+ ion during the flame test. This will allow you to see the light purple K^+ flame or the bright red Li^+ flame if either of the ions is present. You may see colors which correspond to other ions in your unknown. If Ba^{2+} was in your unknown, you may see a brief yellow green flame. Sr^{2+} gives a brief red flame; you should not confuse this with the Li^+ flame because the Sr^{2+} flame is much shorter in duration than the Li^+ flame. Ca^{2+} gives a red-orange colored flame, but it is also much shorter in duration than the Li^+ flame. If K^+ and Li^+ are both present in the unknown, the flame test must be observed carefully, for you will see for 1–2 seconds the light purple color that then turns to the persistent bright red color for Li^+. Since this may be difficult for some to observe, none of the unknowns contain both K^+ and Li^+.</i></p>	<p>Na^+→yellow-orange flame K^+→light purple flame Li^+→bright red flame</p>