

GROUP IV ANALYSIS

(Co^{2+} , Ni^{2+} , Mn^{2+})

EXPERIMENTAL PROCEDURE:

1. Group Precipitation

- Obtain 2-3 ml of Group IV solution or use the decantate from step #1 in Group III.
- Add 3 drops of 3M NH_4Cl and 2 drops of 6M NH_3 to the solution.
- Add 2-3 drops of 20% $(\text{NH}_4)_2\text{S}$ in the fume hood. A precipitate should form at this point.
- Heat the tube in a hot water bath for 10 minutes.
- Centrifuge and decant. Discard the decantate. Save the precipitate for step #2.

2. Separation of Manganese

- Wash the precipitate by adding 10 drops of water. Shake the tube to thoroughly wash the precipitate. Centrifuge and decant. Discard the decantate.
- Add 8 drops of water and 2 drops of 6M HCl to the precipitate. Agitate the solution for one minute, then let set for one minute.
- Centrifuge and decant. Save the decantate for step #3 and the precipitate for step #4.

3. Confirmation of Manganese

- Pour the decantate from step #2C into an evaporating dish. Evaporate to dryness over a low-temperature bunsen burner flame. Do not allow the material to spatter.
- After the material in the evaporating dish has cooled, add 2 drops of 6M HCl and 10 drops of water.
- Add 6 drops of 6M NaOH to the evaporating dish. Pour the mixture into a test tube.
- Centrifuge and decant. Discard the decantate.
- To the precipitate, add 10 drops 9M H_2SO_4 and 10 drops of water. Heat the solution in a hot water bath until the solution boils.
- Add 20 drops of water and a pea-size sample of NaBiO_3 . Shake vigorously. The development of a purple color is a positive test for manganese. If no purple color results complete step G.
- Heat the tube in a hot water bath for 5 minutes. A brownish maroon solution is a positive test for manganese.

(purple also - see flow chart)

4. Separation of Nickel and Cobalt

- A. Wash the precipitate from step #2 C with 10 drops of water. Centrifuge and decant. Discard the decantate.
- B. Add 2 drops of 16M HNO₃ and 6 drops of 12M HCl to the solid in the test tube. Heat the tube in a hot water bath until the solid residue dissolves. (There may be a black substance floating on the top of the liquid. This is sulfur impurities which should be discarded.)
- C. Centrifuge and decant. Discard the solid.
- D. Pour the liquid into an evaporating dish and evaporate to dryness.
- E. After the evaporating dish cools, add 2 drops of 6M HCl and 10 drops of water. Split the solution evenly into two test tubes.

5. Identification of Nickel

- A. To one test tube from step #4E, add drops of 6M NH₃ until the solution is basic.
- B. Add 6 drops of 0.1M DMG. Centrifuge. The formation of a bright red precipitate indicates the presence of nickel.

6. Identification of Cobalt

- A. To the **other** test tube of solution from step #4E, add 5 drops of 1M NH₄SCN and 10 drops of acetone.
- B. If no color results, add 10-15 more drops of acetone.
- C. The formation of a blue solution indicates the presence of cobalt. (If you are doing your unknown and you obtain a red colored solution at this point, add a pea-size sample of ammonium iodide to the solution. If the solution turns blue, cobalt is present.)

Analysis Scheme for Group IV Cations

Group IV Flowchart

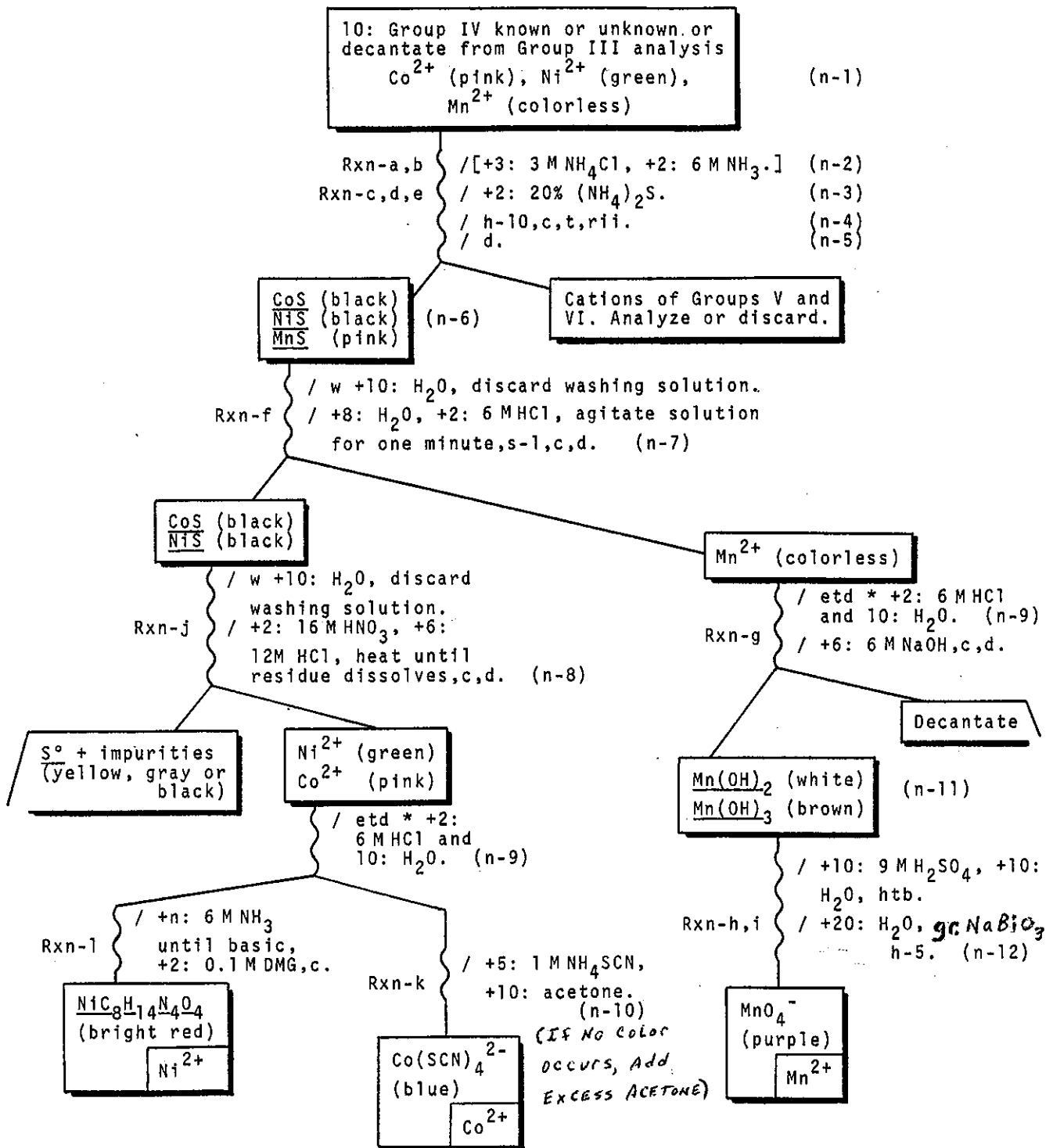


FIGURE 2.5 Group IV flowchart

When doing UNKNOWNs,
if LIQUID TURNS RED
WITH NH₄SCN, Add a
CRYSTAL OF NH₄I + SHAKE
BEFORE ADDING ACETONE.