Sample Procedure

Part A. Separation of Metal Ions

1. Label five small test tubes #1–5. Place about 1 mL (20 drops) of the solution to be analyzed into test tube #1. Record the sample identity and the color of the solution in the data table.
2. Add 5 drops of 3 M HCl and stir to mix. Record the appearance and color of any precipitate.
3. Centrifuge the mixture to separate the solid, if necessary.
4. Test to be sure precipitation is complete: Add one more drop of 3 M HCl to the supernatant. If more precipitate appears, continue adding 3 M HCl dropwise until no more solid forms.
5. Centrifuge and decant (pour off) the supernatant into test tube #2. Alternatively, use a Beral-type pipet to remove the supernatant. Record the color and appearance of the solution in the data table.
6. Save the precipitate in test tube #1 for Part B, step 11.
7. Add 5 drops of 6 M NaOH to the solution in test tube #2.
8. Stir the solution and test with litmus paper to be sure the solution is basic, then add 3 more drops of 6 M NaOH. Record the appearance and color of any precipitate.
9. Centrifuge the mixture to separate the solid, if necessary. Decant the supernatant into test tube #3. Alternatively, use a Beral-type pipet to remove the supernatant. Save the precipitate in test tube #2 for use in Part B, step 13.
10. Record the color and appearance of the solution in test tube #3 and save the solution for use in Part B, step 16.

Part B. Identification of Metal Ions

11. Rinse the precipitate in test tube #1 with 10 drops of distilled water. Tap or swirl the test tube to mix the contents. Centrifuge the mixture and decant the rinse water.
12. Add 8–10 drops of 6 M NH_3 to the solid in test tube #1 and stir to mix. Record observations in the data table.
13. Rinse the precipitate in test tube #2 with 10 drops of distilled water. Tap or swirl the test tube to mix the contents. Centrifuge the mixture and decant the rinse water.
14. Add 5–7 drops of 3 M HCl to the solid in test tube #2 and stir to mix. Test the solution with litmus paper to be sure it is acidic. Record observations in the data table.
15. Add 3 drops of 0.2 M KSCN to the solution in test tube #2 and record observations in the data table.
16. Add 8–10 drops of 3 M HCl to the solution in test tube #3 until the solution tests acidic with litmus paper.
17. Add 3 drops of 0.2 M $K_4Fe(CN)_6$ to the resulting acidic solution in test tube #3 and record observations in the data table.