EXPERIMENT 16E

An Introduction to Qualitative Analysis

The term "qualitative analysis" in chemistry refers to a set of procedures used to identify a particular ion or ions in a given sample when it is not necessary to find out the quantity of any ion present. (Hence the term "qualitative" rather than "quantitative" is used.)

If the number of ions that could be in a sample is large, then the scheme to be followed in order to identify a particular ion correctly becomes very complex. A large number of reagents, many of which may give no result, is required. The quantities and concentrations of reagents involved are critical, since in many cases the separation of two ions depends on relatively small differences in solubility. For these reasons a comprehensive treatment of qualitative analysis is beyond the scope of this lab manual, but it is worthwhile for you to see the methods involved and to acquire enough knowledge of some reactions to enable you to identify some unknowns.

In Part I of this experiment you look at a scheme for identifying different metal ions belonging to Group 2 of the periodic table (the alkaline earth metals), magnesium, calcium, strontium, and barium. Then in Part II you will look at a scheme for identifying four different anions, namely carbonate, sulfate, chloride, and iodide. In each case, after carrying out your reactions, you will be given at least one unknown containing one ion to be identified.

OBJECTIVES

1. to carry out tests on the ions Mg^{2+}, Ca^{2+}, Sr^{2+}, and Ba^{2+} that enable each to be identified separately, and to use these tests to identify an unknown
2. to carry out tests on the ions SO_4^{2-}, CO_3^{2-}, Cl^-, and I^- that enable each to be identified separately, and to use these tests to identify an unknown

MATERIALS

Apparatus

20 test tubes (13 mm × 100 mm)
test-tube rack
lab apron
safety goggles

Reagents

0.1*M* $Mg(NO_3)_2$
0.1*M* $Ca(NO_3)_2$
0.1*M* $Sr(NO_3)_2$
0.1*M* $Ba(NO_3)_2$
0.02*M* K_2CrO_4
0.1*M* $(NH_4)_2C_2O_4$
0.1*M* Na_2SO_4
0.1*M* NaOH
0.1*M* Na_2CO_3
0.1*M* NaCl
0.1*M* NaI
0.1*M* $AgNO_3$
1*M* HNO_3
6*M* NH_3
solution containing unknown cation
solution containing unknown anion

PROCEDURE

Part I Qualitative Analysis of Group 2 Elements

1. Put on your lab apron and safety goggles.
2. Place 2 mL of 0.1*M* $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$, and $Ba(NO_3)_2$ respectively in four 13 mm × 100 mm test tubes.

3. To each tube add 2 mL of 0.02*M* K_2CrO_4, and observe in which tubes a precipitate occurs. Note also the amount of precipitate as light or heavy, and whether it formed immediately or after a short time had elapsed. Record your observations in your copy of Table 1 in your notebook.
4. Repeat Steps 2 and 3, using 2 mL of 0.1*M* $(NH_4)_2C_2O_4$ as the added reagent. Record your observations in Table 1.
5. Repeat Steps 2 and 3, using 2 mL of 0.1 *M* Na_2SO_4 as the added reagent. Again, record what you observe.
6. Repeat Steps 2 and 3, using 2 mL of 0.1*M* NaOH as the added reagent. Record your observations.
7. Obtain an unknown solution containing only one cation, and carry out separate reactions on 2 mL of the sample with 2 mL of each of the four reagents. Identify the cation from your results.
8. Dispose of all the contents of your test tubes according to the reagent disposal instructions. Wash the test tubes, and reuse them in Part II.

CAUTION: Barium compounds are poisonous. Do not get any in your mouth. Do not swallow any.

CAUTION: Chromates are poisonous, and are skin irritants. Do not get any in your mouth; do not swallow any. Wash away any spills and splashes with plenty of water.

CAUTION: Oxalates are poisonous. Do not get any in your mouth. Do not swallow any.

Part II Qualitative Analysis of Selected Anions

1. Place 2 mL of 0.1*M* Na_2CO_3, Na_2SO_4, NaCl, and NaI respectively in four 13 mm × 100 mm test tubes.
2. To each tube, add 2 mL of 1*M* HNO_3. Observe the results, and record them in your copy of Table 2.
3. Repeat Step 1, then add to each tube 2 mL of 0.1*M* $Ba(NO_3)_2$ and note and record in which tubes a precipitate was formed.
4. To the tubes containing precipitates, add 1 mL of 1*M* HNO_3. Observe and record the results.
5. Repeat Step 1, then add to each test tube 2 mL of 0.1*M* $AgNO_3$. Note in which test tubes a precipitate results, and record your results in Table 2.
6. Divide the contents of each test tube containing a precipitate in half, placing each half in a separate test tube.
7. To one set of precipitates add 1 mL of 1*M* HNO_3. Observe the results and record them in Table 2.
8. To the other set of precipitates add 1 mL of 6*M* NH_3, and observe and record the results.
9. Obtain a sample containing a single unknown anion. Carry out each test that you used for the known anions, and observe the results. Identify the anion from your results.
10. Wash your hands thoroughly with soap and water before leaving the laboratory; use a fingernail brush to clean under your fingernails.

CAUTION: Nitric acid is corrosive. Keep it off your skin and out of your eyes. Wash away any spills and splashes with plenty of water.

CAUTION: Silver nitrate is poisonous, and corrosive to skin and eyes. It will result in brown stains on your skin if you spill any on yourself. If this occurs, wash with sodium thiosulfate solution, then with plenty of water. Call your teacher.

CAUTION: Ammonia solution is corrosive. Keep it off your skin and out of your eyes. Avoid breathing its fumes. Wash away any spills and splashes with plenty of water.

REAGENT DISPOSAL

Test tubes containing silver compounds, barium compounds, and chromates should be emptied into the designated waste containers. All other waste material may be safely rinsed down the sink with copious amounts of water.

POST LAB DISCUSSION

The reactions in Part I are all straightforward precipitation reactions. It is important to observe how much precipitate formed, and whether it formed immediately or took somewhat longer to become evident. In some cases these differences are needed to make a definite identification of an unknown.

An important part of the procedure for Part II is adding HNO_3 to see whether the precipitate formed will dissolve in acid. It is quite easy to distinguish between two ions of which both give a precipitate with the same reagent, but one dissolves in acid and the other doesn't.

The $6M$ NH_3 is used to help identify precipitates formed with Ag^+. Some precipitates can dissolve as a result of the formation of the silver diammine ion, $Ag(NH_3)_2^+$; this reaction aids in their identification.

DATA AND OBSERVATIONS

Organize your data and observations in tables similar to the following. It would be a good idea to have these in your notebook before coming to the laboratory.

Part I Qualitative Analysis of Group 2 Elements

Table 1

REAGENTS	0.1 *M* SOLUTIONS OF GROUP 2 CATIONS (AS NITRATES)				
	Mg^{2+}	Ca^{2+}	Sr^{2+}	Ba^{2+}	UNKNOWN # ____
$0.02M$ K_2CrO_4					
$0.1M$ $(NH_4)_2C_2O_4$					
$0.1M$ Na_2SO_4					
$0.1M$ NaOH					

Part II Qualitative Analysis of Selected Anions

Table 2

REAGENTS	0.1 *M* SOLUTIONS OF ANIONS (AS Na SALTS)				
	CO_3^{2-}	SO_4^{2-}	Cl^-	I^-	UNKNOWN # ____
$1M$ HNO_3					
$0.1M$ $Ba(NO_3)_2$					
$0.1M$ HNO_3 added to above					
$0.1M$ $AgNO_3$					
$1M$ HNO_3 added to above					
$6M$ NH_3 added to precipitates from $AgNO_3$					

QUESTIONS

Part I Qualitative Analysis of Group 2 Elements

1. Write net ionic equations for each combination in which a precipitate occurred.
2. State the identity of your unknown (along with its sample number). Give the reasoning you used to arrive at this conclusion.

Part II Qualitative Analysis of Selected Anions

1. Write net ionic equations for each combination in which a precipitate formed or another reaction occurred.
3. Write net ionic equations for each situation in which the precipitate redissolved on the addition of HNO_3 or NH_3.
3. State the identity of your unknown (along with its sample number). Give the reasoning you used to arrive at this conclusion.

FOLLOW-UP QUESTIONS

1. Devise a sequence of reactions to follow (using filtering or centrifuging where necessary to remove precipitates) to identify an unknown containing two or more cations of Group 2 elements.
2. Devise a sequence of reactions to follow (using filtering or centrifuging where necessary to remove precipitates) to identify an unknown consisting of two or more of the anions tested in Part II.
3. Why are the reagents used to test for cations usually alkali metal salts or ammonium salts rather than salts of other metals?
4. Why are the reagents used to test for anions usually a nitrate of the cation that is reacting rather than other salts of that cation?
5. For fast and accurate identification of substances, major research or testing laboratories now use very sophisticated (and expensive) equipment. Find out the name of one of the instruments now used for analysis, and briefly describe its method of operation.

CONCLUSION

State in general terms the principles involved in developing a qualitative analysis scheme.