Experiment 27: Qualitative Analysis of Anions

(This experiment was adapted from Santa Monica College, CHEM 12.)

Purpose

The purpose of this experiment is to use chemical tests to identify common anions in an aqueous solution. This will reinforce the technique of writing and balancing chemical reactions, many of which are acid-base reactions.

Background

In this experiment you will use qualitative analysis to identify the various anions in a sample. Specifically, you will test for the presence of each of the following anions:

CO_3^{2-}, SO_4^{2-}, PO_4^{3-}, SCN^{1-}, Cl^{1-}, NO_3^{1-}

The methodology used in identifying the anions will involve the use of a small portion of the unknown mixture (1 mL) to perform a test for each anion individually.

In some cases, the test for a particular ion will be complicated by the presence of other ions in the mixture that will interfere with the test. In these situations, the interfering ions must be removed before the test can be performed.

Chemicals

1 M Na_2CO_3	0.5 M Na_2SO_4	1 M $BaCl_2$	0.5 M Na_2HPO_4
0.5 M $(NH_4)_2MoO_4$	0.5 M NaSCN	0.1 M $Fe(NO_3)_3$	0.5 M NaCl
0.1 M $AgNO_3$	0.5 M $NaNO_3$	1 M $CuSO_4$	6 M HCl
6 M HNO_3	1 M acetic acid	6 M NaOH	Aluminum granules
Unknown 1	Unknown 2		

Equipment

4 test tubes (rinse & reuse)	glass stirring rod	10-mL grad. cylinder	red litmus paper
dropper pipets	Hot water bath (hood)	blue litmus paper	Centrifuge

General Instructions

For each anion you will perform:

1) a positive control test on 1 mL of solution of the anion
2) a positive control test on 1 mL of a diluted solution of the anion. (The diluted anion solutions can be prepared by adding 10 drops of deionized water to 10 drops of the anion solution. This will yield approximately 1 mL of a diluted solution.)
3) the same test on 1 mL of each unknown solution, which will contain at least one of the anions in the control solutions.

Record the procedure steps and observations in your notebook. Tabulate your observations using the following format:

Test Tube #	1	2	3	4
Anion being Tested	Control Test Observation	Diluted Control Test Observation	Unknown Solution 1	Unknown Solution 2

The specific anion tests are:

Test for the presence of carbonate ion, CO_3^{2-}

1) Add 1 mL of 1 M Na_2CO_3 (the positive control) and 1 mL of 6 M HCl to a test tube. Effervescence indicates the presence of CO_3^{2-}. In the concentrated control solution, you should see effervescence for at least a few seconds.
2) Repeat this test with the diluted control solution. It may be necessary to place the test tube in the hot water bath (in the hood) in order to observe the effervescence.
3) Repeat this test with each unknown. If no effervescence is observed, place the test tubes in the hot water bath before concluding whether CO_3^{2-} is present or not.

Test for the presence of sulfate ion, SO_4^{2-}

1) Add 1 mL of 0.5 M Na_2SO_4 (the positive control) and 1 mL of 6 M HCl to a small test tube. Next add 3 drops of 1 M $BaCl_2$. A finely divided, white precipitate indicates the presence of the SO_4^{2-} ion.
2) Repeat this test with the diluted control solution.
3) Repeat this test with each unknown. Use your observations to conclude whether SO_4^{2-} is present or not in each unknown.

Test for the presence of phosphate ion, PO_4^{3-}

1) Add 1 mL of 0.5 M Na_2HPO_4 (the positive control) and 1 mL of 6 M HNO_3 to a small test tube. Next add 1 mL of 0.5 M $(NH_4)_2MoO_4$ and **stir thoroughly**. Place the test tube in the hot water bath and continue to stir. <u>This test tube **MUST** be heated.</u> A yellow precipitate of indicates the presence of PO_4^{3-}.
2) Repeat this test with the diluted control solution.
3) Repeat this test with each unknown. Use your observations to conclude whether PO_4^{3-} is present or not in each unknown. Clear yellow is not a precipitate. A precipitate makes a mixture cloudy.

This is the likely chemical equation for this reaction:

$$8HNO_3 + 2Na_3PO_4 + 4(NH_4)MoO_4 \rightarrow 4H_2O + 6NaNO_3 + 2NH_4NO_3 + 2(NH_4)_3PO_4 + 4MoO_{3(s)}$$

Test for the presence of thiocyanate ion, SCN^{1-}

1) Add 1 mL of 0.5 M NaSCN (the positive control) and 1 mL of 1 M acetic acid to a small test tube. Now add 2 drops of 0.1 M $Fe(NO_3)_3$. An orange/red solution indicates the presence of SCN^{1-} (as $FeNCS^{2+}{}_{(aq)}$)
2) Repeat this test with the diluted control solution.
3) Repeat this test with each unknown. Use your observations to conclude whether SCN^{1-} is present or not in each unknown.

Important: If your unknown contains PO_4^{3-} it will interfere with the test for the SCN^{1-} since PO_4^{3-} will form a precipitate with Fe^{3+} (white, greyish, or pink solid).

$$Fe^{3+}{}_{(aq)} + PO_4^{3-}{}_{(aq)} \rightarrow FePO_{4(s)}$$

In this case it will be necessary to remove all of the PO_4^{3-} before any conclusion can be made concerning the presence of SCN^{1-}. The PO_4^{3-} (as $FePO_{4(s)}$) can be removed by centrifuging the mixture and decanting the supernatant solution. Now add $Fe(NO_3)_3$ to the supernatant solution. If more precipitate forms, centrifuge and decant a second time. Now test for the presence of thiocyanate ion by adding the $Fe(NO_3)_3$ to the supernatant solution. A dark red solution indicates the presence of SCN^{1-}.

Test for the presence of chloride ion, Cl^{1-}

1) Add 1 mL of 0.5 M NaCl (the positive control) and 1 mL of 6 M HNO_3 to a small test tube. Next add 2 to 3 drops of 0.1 M AgNO3. The formation of a white, curdy precipitate indicates the presence of Cl^{1-}. *(Curdy means small lumps.)*
2) Repeat this test with the diluted control solution.
3) Repeat this test with each unknown. Use your observations to conclude whether Cl^{1-} is present or not in each unknown.

Important: If your unknown contains SCN^{1-} it will interfere with the test for the Cl^{1-} since it will form a white precipitate with Ag^{1+}.

$$Ag^{1+}{}_{(aq)} + SCN^{1-}{}_{(aq)} \rightarrow AgSCN_{(s)}$$

In this case put 1 mL of your unknown sample into a small 50 mL beaker and add 1 mL of 6 M HNO_3. Boil the solution very gently on the hotplate (in the hood) until the volume has decreased by about one half. Under these conditions the SCN^{1-} will decompose. Now pour this solution into a small test tube, and test for the presence of the Cl^{1-} ion by adding 1 mL of 6 M HNO_3 and 2 to 3 drops of 0.1 M $AgNO_3$. A white curdy precipitate indicates the presence of Cl^{1-}.

Test for the presence of nitrate ion, NO_3^{1-}

1) Add 1 mL of 0.5 M $NaNO_3$ (the positive control) and 1 mL of 6 M NaOH to a small test tube. Then add a few granules of aluminum metal and put the test tube in the hot water bath (in the hood). The reaction between Al and NaOH will produce H_2 gas which will reduce the NO_3^{1-} to NH_3. The NH_3 can be detected by placing a piece of moistened red litmus paper directly above (but not in contact with) the mouth of the test tube. If the red litmus paper turns blue (due to NH_3 vapor coming out of the test tube) then it can be concluded that NO_3^{1-} is present in the unknown. Note that small blue spots produced on the red litmus paper are the result of spray from the basic solution in the test tube and do not necessarily indicate the presence of nitrate.
2) Repeat this test with the diluted control solution.
3) Repeat this test with each unknown. Use your observations to conclude whether NO_3^{1-} is present or not in each unknown.

   This test is referred to as Devarda's test (Chemistry.Stackexchange.com):
   $3\ NO_3^{1-}{}_{(aq)} + 8\ Al_{(s)} + 5\ OH^{1-}{}_{(aq)} + 18\ H_2O_{(l)} \rightarrow 3\ NH_{3(g)} + 8\ [Al(OH)_4]^{1-}{}_{(aq)}$

Important: If your unknown contains SCN^{1-} it will interfere with the test for the NO_3^{1-}. SCN^{1-} will give a false-positive result for NO_3^{1-}. In this case add 1 mL of your unknown to 1 mL of 1 M $CuSO_4$ in a small test tube. Place the test tube in a hot water bath for about 2 minutes (in the hood). Centrifuge the mixture and decant the supernatant solution into another small test tube. The solid may be discarded in the waste.
To the supernatant solution add 1 mL of 1 M Na_2CO_3. Centrifuge the mixture and decant 1 mL of the supernatant solution into another small test tube. The solid may again be discarded in the waste.
Now test for the presence of nitrate ion by using the supernatant solution in the test tube and adding 1 mL of 6 M NaOH and a few granules of aluminum metal. Place the test tube in the hot water bath and use red litmus paper to test for the presence of NO_3^{1-} as described in step 1 above.

In the Data Tables section of your notebook:

1) Tabulate your results for the control test, diluted control test, unknown 1 and unknown 2. Use the following format.

Anion being Tested	Control Test Observation	Diluted Control Test Observation	Unknown Solution 1 Obs.	Unknown Solution 2 Obs.

In the Calculations / Results section of your notebook:

1) Use the chart on the next page for your two unknowns that includes all of the anion tests performed and your observations and conclusion about each unknown for each test. Cut-out and tape this chart into your notebook.
2) Write the balanced chemical equation for the carbonate test, sulfate test, thiocyanate test, and chloride ion test. (4 points total; 1 point per equation)

In the Conclusion section of your notebook:

1) Summarize the anions present in each of your two unknowns and the logic used to make your decisions.

Please use this chart in the Calculations / Results section of your notebook. Cut-out and tape this chart into your notebook.

Anion Test Performed	Unknown 1	Unknown 2
Test for CO_3^{2-} Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Test for SO_4^{2-} Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Test for PO_4^{2-} Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Test for SCN^- Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Test for Cl^- Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Test for NO_3^- Observation ------------------ Conclusion (1 point)	-----------------------------------	-----------------------------------
Overall Conclusion: List anions present in each unknown (2 points)		

Do not include information about interfering ion work.
Each conclusion for each unknown in the table above is worth 1 point.
Each overall conclusion is worth 2 points.
The table above is worth a possible 16 points.