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# Qualitative Analysis of Anions using Spot Tests

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## Objectives

The objectives of this laboratory are to use spots tests to identify common anions in an aqueous solution.

## Background

In the previous two experiments, you have used qualitative analysis to determine the identity of various cations in a sample. 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:  $\text{CO}_3^{2-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ ,  $\text{SCN}^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ .

The methodology used in identifying the anions will be different than that which was used to identify cations. Here you will use a small portion of the unknown mixture (1 mL) to perform a spot 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.

## Procedure

### Chemicals

1 M  $\text{Na}_2\text{CO}_3$ , 0.5 M  $\text{Na}_2\text{SO}_4$ , 1 M  $\text{BaCl}_2$ , 0.5 M  $\text{Na}_2\text{HPO}_4$ , 0.5 M  $(\text{NH}_4)_2\text{MoO}_4$ , 0.5 M  $\text{KSCN}$ , 0.1 M  $\text{Fe}(\text{NO}_3)_3$ , 0.5 M  $\text{NaCl}$ , 0.1 M  $\text{AgNO}_3$ , 0.5 M  $\text{NaNO}_3$ , 1 M  $\text{CuSO}_4$ , 6 M  $\text{HCl}$ , 6 M  $\text{HNO}_3$ , 6 M acetic acid, 6M  $\text{NaOH}$ , Aluminum granules

### Equipment

8 small test tubes, glass stirring rod, small 10-mL graduated cylinder, 250-mL beaker, stand, ring clamp, wire gauze, small watch glass, dropper pipets, blue litmus paper, red litmus paper, wash bottle filled with deionized water, Bunsen burner, centrifuge

## Safety and Waste Disposal

1. All waste generated by this lab is toxic and must be disposed of in the hazardous waste container!
2. Unused sample of the unknown should be discarded in the hazardous waste container. Wash the empty test tube and return it to your instructor's bench.
3. There are quite a few concentrated acids and bases in this experiment. Please be careful when handling them.

## General Instructions

For each anion you will perform a positive control test on 1 mL of solution of the anion, followed by a positive control experiment on 1 mL of a diluted solution of the anion. The diluted anion solutions can be prepared by adding 3 drops of deionized water to 9 drops of the anion solution. This will yield approximately 1 mL of a diluted solution.

You will then perform the same test on 1 mL of your unknown solution which may or may not contain the anion in the control solution.

### Test for the presence of carbonate ion, $\text{CO}_3^{2-}$

1. Add 1 mL of 1M  $\text{Na}_2\text{CO}_3$  (or your unknown) and 1 mL of 6M HCl to a small test tube. Effervescence indicates the presence of  $\text{CO}_3^{2-}$ . In the concentrated control solution you should see effervescence for at least a few seconds. In the diluted control solution it may be necessary to place the test tube in a hot water bath in order to observe the effervescence. For the unknown if no effervescence is observed, place the test tube in the hot water bath before concluding that  $\text{CO}_3^{2-}$  is not present.

### Test for the presence of sulfate ion, $\text{SO}_4^{2-}$

2. Add 1 mL of 0.5 M  $\text{Na}_2\text{SO}_4$  (or your unknown) and 1 mL of 6 M HCl to a small test tube. Next add a few drops of 1 M  $\text{BaCl}_2$ . A finely divided, white precipitate indicates the presence of the  $\text{SO}_4^{2-}$  ion.

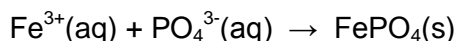
### Test for the presence of phosphate ion, $\text{PO}_4^{3-}$

3. Add 1 mL of 0.5 M  $\text{Na}_2\text{HPO}_4$  (or your unknown) and 1 mL of 6 M  $\text{HNO}_3$  to a small test tube. Next add a 1 mL of 0.5 M  $(\text{NH}_4)_2\text{MoO}_4$  and **stir thoroughly**. Place the test tube in the hot water bath and continue to stir. A yellow precipitate indicates the presence of  $\text{PO}_4^{3-}$ .

**Test for the presence of thiocyanate ion, SCN<sup>-</sup>**

4. Add 1 mL of 0.5 M KSCN (or your unknown) and 1 mL of 6 M acetic acid (HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>) to a small test tube. Now add 2 drops of 0.1 M Fe(NO<sub>3</sub>)<sub>3</sub>. A dark red solution indicates the presence of SCN<sup>-</sup>.

**Important:** If your unknown contains PO<sub>4</sub><sup>3-</sup> it will interfere with the test for the SCN<sup>-</sup> since it will form a precipitate with Fe<sup>3+</sup>.

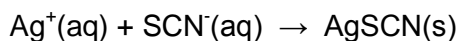


In this case it will be necessary to remove all of the PO<sub>4</sub><sup>3-</sup> before any conclusion can be made concerning the presence of SCN<sup>-</sup>. The PO<sub>4</sub><sup>3-</sup> can be removed by centrifuging the mixture and decanting the supernatant solution. Now add Fe(NO<sub>3</sub>)<sub>3</sub> to the supernatant solution. If more precipitate forms, centrifuge and decant a second time. Add the Fe(NO<sub>3</sub>)<sub>3</sub> to the supernatant solution. A dark red solution indicates the presence of SCN<sup>-</sup>.

**Test for the presence of chloride ion, Cl<sup>-</sup>**

5. Add 1 mL of 0.5 M NaCl (or your unknown) and 1 mL of 6 M HNO<sub>3</sub> to a small test tube. Next add 2 to 3 drops of 0.1 M AgNO<sub>3</sub>. The formation of a white, curdy precipitate indicates the presence of Cl<sup>-</sup>.

**Important:** If your unknown contains SCN<sup>-</sup> it will interfere with the test for the Cl<sup>-</sup> since it will form a white precipitate with Ag<sup>+</sup>.



In this case put 1 mL of your unknown sample into a small beaker (30 mL or 50 mL) and add 1 mL of 6 M HNO<sub>3</sub>. Boil the solution very gently (directly over the open flame) until the volume has decreased by about one half. Under these conditions the SCN<sup>-</sup> will decompose. Now pour this solution into a small test tube, add 1 mL of 6M HNO<sub>3</sub> and 2 to 3 drops of 0.1 M AgNO<sub>3</sub>. A white curdy precipitate indicates the presence of Cl<sup>-</sup>.

**Test for the presence of nitrate ion,  $\text{NO}_3^-$** 

6. Add 1 mL of 0.5 M  $\text{NaNO}_3$  (or your unknown) and 1 mL of 6 M  $\text{NaOH}$  to a small test tube. Then add a few granules of aluminum metal and put the test tube in the hot water bath. The reaction between Al and  $\text{NaOH}$  will produce  $\text{H}_2$  gas which will reduce the  $\text{NO}_3^-$  to  $\text{NH}_3$ . The  $\text{NH}_3$  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 uniformly blue (due to  $\text{NH}_3$  vapor coming out of the test tube) then it can be concluded that  $\text{NO}_3^-$  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.

**Important:** If your unknown contains  $\text{SCN}^-$  it will interfere with the test for the  $\text{NO}_3^-$ . In this case add 1 mL of your unknown to 1 mL of 1 M  $\text{CuSO}_4$  in a small test tube. Place the test tube in a hot water bath for about 2 minutes. 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  $\text{Na}_2\text{CO}_3$ . 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.

To the supernatant solution in the test tube add 1 mL of 6 M  $\text{NaOH}$  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  $\text{NO}_3^-$  as described in the previous step.

Lab Partner: \_\_\_\_\_

Lab Section: \_\_\_\_\_

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**Lab Report: Qualitative Analysis of Anions using Spot Tests**

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Indicate your observations in the table below. Based on your observations indicate which anions are present in your unknown sample.

Ion	Control	Diluted Control	Unknown
$\text{CO}_3^{2-}$			
$\text{SO}_4^{2-}$			
$\text{PO}_4^{3-}$			
$\text{SCN}^-$			
$\text{Cl}^-$			
$\text{NO}_3^-$			

Unknown number \_\_\_\_\_

Ions present in your unknown \_\_\_\_\_