Chemistry 117 Laboratory
University of Massachusetts, Boston

QUALITATIVE ANALYSIS OF ANIONS

LEARNING GOALS

1. Become an expert at writing net ionic equations.
2. Obtain exposure to deductive reasoning as a scientific approach.

TASK

In this lab you will perform standard tests that are used to confirm the presence of different anions in solution. After gaining some experience with these tests and briefly analyzing your results, you will devise a strategy to test three unknown solutions to determine the major anion present in each.

INTRODUCTION

This task is similar the experiment performed last week. In Parts 1-3 you will gain some experience with standard tests using known solutions and making observations. Then you will conduct these several of these standard tests to confirm the presence of one of six possible anions in a three unknown solutions. A summary of how each of these six anions reacts in these standard tests is provided below.

A. Carbonate Reactions (CO_3^{2-})

A1. Aqueous sodium carbonate and sulfuric acid react with the evolution of a colorless, odorless gas ($CO_{2(g)}$).
A2. Aqueous sodium carbonate forms a white precipitate in aqueous barium Chloride ($BaCO_{3(s)}$).
A3. When aqueous nitric acid is added to the precipitate in A2, the precipitate dissolves and a colorless, odorless gas is evolved ($CO_{2(g)}$).

B. Nitrate Reactions (two consecutive reactions B1 and B2)

B1. Aqueous sodium nitrate and $Fe^{2+}_{(aq)}$ react in acidic solution forming $Fe^{3+}_{(aq)}$ and a colorless, nitric oxide ($NO_{(g)}$) gas.
B2. Nitric oxide is immediately oxidized to the red-brown nitrogen dioxide gas by molecular oxygen in the air.

C. Nitrite Reactions

C1. Aqueous sodium nitrite disproportionates to nitric oxide gas and aqueous nitrate ion in warm acidic solution.
C2. On contact with the air nitric oxide is oxidized to a red-brown gas.

D. Phosphate Reactions

D1. Aqueous sodium phosphate forms a white precipitate ($Ba_3(PO_4)_{2(s)}$) in aqueous barium chloride.
D2. When aqueous nitric acid is added to the precipitate formed in D1, the precipitate dissolves.
D3. When aqueous ammonium molybdate, $(NH_4)_2MoO_4$, is added to aqueous sodium phosphate in acid solution, a bright yellow precipitate of ammonium molybdophosphate, $(NH_4)_3PMo_{12}O_{40}$, forms.

E. Sulfate Reaction

E1. Aqueous sodium sulfate forms a white precipitate ($BaSO_4$) in aqueous barium chloride.

F. Sulfite Reactions

F1. When aqueous sodium sulfite is acidified with sulfuric acid, a colorless gas with a choking odor ($SO_{2(g)}$) is evolved.

F2. Aqueous sodium sulfite forms a white precipitate ($BaSO_{3(s)}$) in aqueous barium chloride.

F3. When the precipitate formed in reaction F2 is acidified with nitric acid, the precipitate dissolves and a colorless gas with a choking odor ($SO_{3(g)}$) is evolved.

Procedure

Perform each of the following tests in a clean, small (75mm) test tube. Take only a tiny bit of solid (about the size of a grain of rice) for these tests. WARNING! If you take too much, some solids which are expected to dissolve in the prescribed amount of reagent will not do so. Set up a warm water bath for use in several of the tests. Use a beaker of water on a hotplate with the temperature control set as low as possible.

Carry out parts 1 and 2 for each known solid in turn. *Work with only a single unkown at a time.*

Part 1. Barium solubility tests on each of the six known solids

- Label six clean and dry test tubes according to the six anions being tested.
- Place a tiny bit of each of the known sodium salts and 10 drops of deionized water into the appropriately labeled test tube. Swirl the each test tube until the solid dissolves.
- To each test tube, add 5 drops of $BaCl_2$ solution, swirl, and look for a precipitate. If a precipitate forms, write this observation (Table, Part 1) and the net ionic equation for the reaction in the Table under "Add (aq)." However, if you do not detect a $BaCl_2$ precipitate, enter "NR" (no reaction) in the Table, skip the next test and go directly to Part 2.
- If a precipitate forms, add 5 drops of HNO_3 to the same test tube, swirl and watch what happens. Note the color and odor of any gas that might evolve. USE CAUTION IN SNIFFING THE GAS. Never take a big whiff, but waft the gas toward your nose by moving your hand over the tube creating an air stream containing the gas. If you see no bubbles and detect no odor, gently warm the test tube in a warm water bath to increase the evolution rate of any gas that might be produced. If still no gas evolves, write "no rxn" under the "then add HNO_3" column.
- Empty the test tubes in the waste container in the hood and obtain six new test tubes for Part 2.

Part 2. Volatile products tests on each of the six known solids

- Label six clean and dry test tubes according to the six anions being tested.
- Place a tiny bit of each of the known sodium salts and 5 drops of H_2SO_4 into the appropriately labeled test tube. PERFORM THE TEST ON EACH TEST TUBE

ONE AT A TIME, so you can make your observations. Watch what happens. Note the color and odor of any gas that might evolve.

- If you see no bubbles and detect no odor, gently warm the test tube in a warm water bath to increase the evolution rate of any gas that might be produced.
- Write your observations and the net ionic equation or equations for any reaction(s) evolving gas in the TABLE for Part 2 on the Answer Sheet.

Part 3. Confirmatory reactions for nitrate and phosphate only

Carry out the following special reactions for nitrate and phosphate knowns. These tests may help you identify if your unknown contains one of these ions.

Nitrate Confirmatory Test

- Add a tiny amount of sodium nitrate to a clean test tube. Add 5 drops of $FeSO_4$ reagent and gently swirl. Carefully warm the test tube in a hot water bath. CAUTION! Gas may evolve rapidly. DO NOT POINT THE TEST TUBE AT ANYBODY. Write your observations and the net ionic equations on the Report Sheet.

Phosphate Confirmatory Test

- Take a tiny amount of known sodium phosphate and dissolve in water. Then take 10 drops of HNO_3 and 4 drops of $(NH_4)_2MoO_4$ solution in a clean test tube. A precipitate will usually form. If the precipitate does not immediately appear warm the test tube. Record your observations and net ionic equation on the Answer Sheet (Part 3).

Part 4. Analysis of three unknowns

You will be assigned three unknowns which are sodium salts of the six anions studied above. Carry out as many tests as you wish in order to identify the unknowns. The following information is then recorded in Part 4 (Unknown Analysis) of the Answer Sheet. For each unknown record the ID number, a list of tests performed, your observations for each test, and your conclusions as to what anions the test eliminates or confirms.

Name _____ Section _____

Part 1. BARIUM SOLUBILITY TESTS

Add $BaCl_{2(aq)}$, then add $HNO_{3(aq)}$

CO_3^{2-}	White precipitate forms $CO_3^{2-} + Ba^{2+} \rightarrow BaCO_{3(s)}$	Bubbles of gas evolved as precipitate dissolves $3BaCO_{3(s)} + 2H^+ \rightarrow CO_{2(g)} + Ba^{2+} + H_2O$
NO_3^-		
NO_2^-		
PO_4^{3-}		
SO_4^{2-}		
SO_3^{2-}		

Write the net ionic equation for one of the above reactions.

Part 2. VOLATILE PRODUCTS TESTS

Add $H_2SO_{4(aq)}$

CO_3^{2-}
NO_3^-
NO_2^-
PO_4^{3-}
SO_4^{2-}
SO_3^{2-}

Use A-F in the introduction to help you identify the gas that evolved.

Write the net ionic equation for one of the above reactions.

Part 3. CONFIRMATORY REACTIONS

Nitrate test

Observations: _____

_____

Net Ionic Equation: _____

Phosphate test

Observations: _____

_____

Net Ionic Equation: _____

Part 4. UNKNOWN ANALYSES

Unknown 1: Sample number _____ Identify anion(s) present _____

Describe how you came to this conclusion. _____

_____

_____

_____

Unknown 2: Sample number _____ Identify anion(s) present _____

Describe how you came to this conclusion. _____

_____

_____

_____

Unknown 3: Sample number _____ Identify anion(s) present _____

Describe how you came to this conclusion. _____

_____

_____

Practice with net ionic equations (OPTIONAL)

Write net ionic equations (NIE) on the fourteen reactions in the introduction; A1-A3, B1, B2, C1, C2, D1-D3, E1 and F1-F3.

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D2. When aqueous nitric acid is added to the precipitate formed in D1, the precipitate dissolves.

D3. When aqueous ammonium molybdate, $(NH_4)_2MoO_4$, is added to aqueous 4 sodium phosphate in acid solution, a bright yellow precipitate of ammonium molybdophosphate, $(NH_4)_3PMo_{12}O_{40}$, forms.
Hint: This is not a redox reaction and reactants are NH_4^+, MoO_4^{2-}, H_3PO_4 and H^+. Balance by inspection by adding H_2O to either side as required.

E. Sulfate Reaction

E1. Aqueous sodium sulfate forms a white precipitate ($BaSO_4$) in aqueous barium chloride.

F. Sulfite Reactions

F1. When aqueous sodium sulfite is acidified with sulfuric acid, a colorless gas with a choking odor ($SO_{2(g)}$) is evolved.

F2. Aqueous sodium sulfite forms a white precipitate ($BaSO_{3(s)}$) in aqueous barium chloride.

F3. When the precipitate formed in reaction F2 is acidified with nitric acid, the precipitate dissolves and a colorless gas with a choking odor ($SO_{3(g)}$) is evolved.

LAB REPORT

There will be no abstract required for this experiment. Your lab report will consist of your data sheets (pg 3 and 4) and a summary of your identifications. The data sheet is worth 30 points.

The Summary: Identify each of your unknown sodium salts in Part 4. Describe the tests that were performed and the interpretation of the results of these tests that enabled your identifications. There are three unknowns, so you will receive four points for the correct identification of each unknown sodium salt, three points each for the outline of the tests that were performed on each unknown salt and three points for the correct explanation of how you were able to use your experimental observations to deduce the identification of each of unknown salts.