

Name _____ Section _____

Chemistry 104 Laboratory
University of Massachusetts, Boston

QUALITATIVE ANALYSIS OF ANIONS

PRELAB ASSIGNMENT

Write net ionic equations (NIE) on the fourteen blank lines on this and the next page.

This experiment uses techniques of qualitative analysis to identify unknown salts based on the anion species. In contrast to the cation qualitative analysis schemes done previously, the unknowns here will be solid samples rather than solutions and each sample will consist of a single salt rather than a mixture. The unknowns will be sodium salts of the following anions: carbonate, nitrate, nitrite, phosphate, sulfate and sulfite. The test procedures for determining the identities of the unknown are based on the chemistry of these anions as shown by the following reactions.

A. Carbonate Reactions

A1. Aqueous sodium carbonate and sulfuric acid react with the evolution of a colorless, odorless gas.

NIE: _____

A2. Aqueous sodium carbonate forms a white precipitate in aqueous barium chloride.

NIE: _____

A3. When aqueous nitric acid is added to the precipitate in A2, the precipitate dissolves and a colorless, odorless gas is evolved.

NIE: _____

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

B1. Aqueous sodium nitrate and $\text{Fe}^{2+}(\text{aq})$ react in acidic solution forming colorless nitric oxide gas and $\text{Fe}^{3+}(\text{aq})$.

NIE: _____

B2. Nitric oxide is immediately oxidized to the red-brown nitrogen dioxide gas by molecular oxygen in the air.

NIE: _____

C. Nitrite Reactions (two consecutive reaction C1 and C2)

C1. Aqueous sodium nitrite disproportionates to nitric oxide gas and aqueous nitrate ion in warm acidic solution.

NIE: _____

C2. On contact with the air nitric oxide is oxidized to a red-brown gas.

NIE: _____

D. Phosphate reactions

D1. Aqueous sodium phosphate forms a white precipitate in aqueous barium chloride.

NIE: _____

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

NIE: _____

D3. When aqueous ammonium molybdate, $(\text{NH}_4)_2\text{MoO}_4$, is added to aqueous sodium phosphate in acid solution, a bright yellow precipitate of ammonium molybdophosphate, $(\text{NH}_4)_3\text{PMo}_{12}\text{O}_{40}$, 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.

NIE: _____

E. Sulfate Reaction

E1. Aqueous sodium sulfate forms a white precipitate in aqueous barium chloride.

NIE: _____

F. Sulfite Reactions

E1. When aqueous sodium sulfite is acidified with sulfuric acid, a colorless gas with a choking odor is evolved.

NIE: _____

F2. Aqueous sodium sulfite forms a white precipitate in aqueous barium chloride.

NIE: _____

F3. When the precipitate formed in reaction F2 is acidified with nitric acid, the precipitate dissolves and a colorless gas with a choking odor is evolved.

IN THE LABORATORY

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 unknown at one time.*

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

Add a tiny bit of solid and 10 drops of deionized water to the test tube. Swirl the test tube until the solid dissolves.

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 $\text{BaCl}_2(\text{aq})$.” However, if you do not detect a 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. Record your observation in the Table (then, add HNO_3 column).

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

Add a tiny amount of solid and 5 drops of H_2SO_4 to a clean test tube. 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 you 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, 10 drops of HNO_3 and 4 drops of $(\text{NH}_4)_2\text{MoO}_4$ 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.

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Part 1. BARIUM SOLUBILITY TESTS

Add BaCl₂(aq)

Then add HNO₃(aq)

CO ₃ ²⁻	white precipitate forms Ba ²⁺ + CO ₃ ²⁻ = BaCO ₃ (s)	bubbles of gas evolved as precipitate dissolves BaCO ₃ (s) + 2H ⁺ = Ba ²⁺ + CO ₂ (g) + H ₂ O
NO ₃ ⁻		
NO ₂ ⁻		
PO ₄ ³⁻		
SO ₄ ²⁻		
SO ₃ ²⁻		

Part 2. VOLATILE PRODUCTS TESTS

Add H₂SO₄(aq)

CO ₃ ²⁻	
NO ₃ ⁻	
NO ₂ ⁻	
PO ₄ ³⁻	
SO ₄ ²⁻	
SO ₃ ²⁻	

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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. _____

