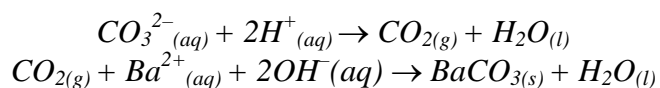


## II. QUALITATIVE ANALYSIS OF ANIONS

Obtain both the anion standards sample and the unknown anions sample. As with the analysis of cations, record the results of each step in the Anion Data Table.

### 1. Confirmation of Carbonate.

*In acid solution, carbonate forms carbon dioxide gas and water. The carbon dioxide may be seen as a slight effervescence. Carbon dioxide is less soluble in hot water than cold water. When carbon dioxide gas is passed through a saturated solution of barium hydroxide, it readily forms a precipitate of white barium carbonate.*



*If any bubbles were formed when acid was added to the original solution, carbonate is probably present and carbon dioxide is being formed. A confirmation of the presence of carbonate involves reacting evolving carbon dioxide with barium hydroxide to form white, insoluble barium carbonate.*

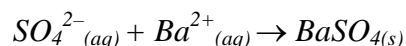
Prepare the agent = 2 mL of clear, saturated Ba(OH)<sub>2</sub> solution (“baryte water”) in a test tube to be available for the test with carbon dioxide.

- Place 1 mL of the original test solution (or unknown solution) in a test tube.
- Acidify this solution by adding 0.5 mL of 6 M HNO<sub>3</sub>.
- Place the tube in a hot water bath and observe to see if any gas bubbles form.
- Take a dry **Beral-type pipette** (see the picture below) and squeeze the bulb closed. Place the tip of the pipette close to (but not touching) the surface of the liquid in the test tube and slowly release the bulb to draw escaping **carbon dioxide into the pipette**.
- Put the pipette into the barium hydroxide solution, and slowly squeeze the bulb, causing the gas in the pipette to bubble through the barium hydroxide solution. This procedure may be repeated. The formation of a cloudy white precipitate of barium carbonate confirms the presence of carbonate ion in the original solution.



## 2. Confirmation of Sulfate.

*The test for sulfate is the formation of white, insoluble barium sulfate. This solid is insoluble even in acidic solution.*

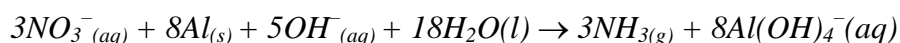


- Place 0.5 mL of the original test solution (or unknown solution) in a test tube.
- Add 6 M nitric acid,  $\text{HNO}_3$ , dropwise until the solution is acidic.
- Add 0.5 mL 0.1 M  $\text{BaCl}_2$  solution. The formation of a white precipitate of  $\text{BaSO}_4$  confirms the presence of sulfate.

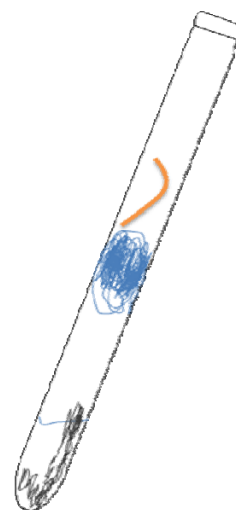
## 3. Confirmation of Nitrate.

*The test for nitrate involves the reduction of nitrate ions in a basic solution to ammonia,  $\text{NH}_3$ , using solid aluminum as the reducing agent. When the solution is heated, ammonia gas is liberated.*

*The evolving ammonia gas will turn pH paper yellow to dark green.*



- Place 1 mL of the original test solution (or unknown solution) in a test tube.
- Add 10%  $\text{NaOH}$  dropwise until the solution is basic, and then add 6 drops in excess.
- Add the tip of a spatula containing **aluminum granules**.
- Place a **small paper/cotton wad** loosely about halfway down the test tube, but not touching the solution. This is to prevent spattering of the solution onto the pH paper.
- Hang a piece of **moist pH paper** in the tube so that the bottom of the paper is close to the wad.
- Warm the solution in a hot water bath (or over a burner) until it starts bubbling strongly. Be sure that the solution do not touch the pH paper.
- Allow the solution to cool. A slow color change (within 3 to 5 minutes) of the pH paper to blue, starting at the bottom and spreading to the top, indicates the evolution of ammonia and confirms the presence of nitrate ion in the original solution.



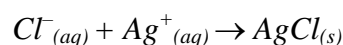
## Precipitation of most of the interfering cations prior to analysis of other anions



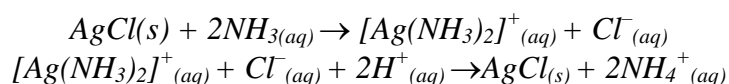
To the sample on a porcelain **evaporating dish**, add solution of sodium carbonate until precipitation occur and the solution is basic (check by a pH paper). Then boil for 5 min and filtrate. Dispose of the precipitation on the filter, **keep the filtrate** and **neutralize** the filtrate with nitric acid (15%).

### 4. Separation of the Chloride ( $\text{Cl}^-$ ); Confirmation of Chloride.

*Chloride ion forms an insoluble silver compound. Silver chloride is a white solid.*



*Silver chloride dissolves in 6 M ammonia,  $\text{NH}_3$ , forming the colorless ion  $[\text{Ag}(\text{NH}_3)_2]^+$ . If nitric acid,  $\text{HNO}_3$ , is added to a solution containing this ion, the ammonia in the complex reacts with hydrogen ions to form ammonium ions, and the silver recombines with the chloride ions that are still present in solution.*



- Place 1 mL of the filtrate in a test tube.
- Add 1 mL of 0.1 M silver nitrate,  $\text{AgNO}_3$ . A precipitate of  $\text{AgCl}$  will form.
- Filtrate the mixture, wash the solid with 1 mL distilled water, and **pour off the filtrate**.
- “Wash” the precipitate with 1 mL 6 M ammonia,  $\text{NH}_3$ , to dissolve the precipitate  $\text{AgCl}$ . Catch the filtrate which will contain chloride ion.
- Add 1 mL 6 M nitric acid,  $\text{HNO}_3$ , to the filtrate containing the dissolved silver chloride.
- The solution will get hot and smoke from the reaction with the excess ammonia whether or not silver chloride is present.
- Test with pH paper to see if the solution is acidic. If it is not, add more  $\text{HNO}_3$  until the solution is acidic. The appearance of the white precipitate of  $\text{AgCl}$  in the acidic solution confirms the presence of chloride.

### 5. Repeat anion procedures 1–4 for the anion unknown sample.

### ANION ANALYSIS DATA TABLE

**Known Solution**

**Unknown Solution**

| <b>Step</b> | <b>Procedure</b> | <b>Results</b> | <b>Conclusion</b> | <b>Results</b> | <b>Conclusion</b> |
|-------------|------------------|----------------|-------------------|----------------|-------------------|
| 1           |                  |                |                   |                |                   |
| 2           |                  |                |                   |                |                   |
| 3           |                  |                |                   |                |                   |
| 4           |                  |                |                   |                |                   |