Qualitative Analysis
Identification of Some Important Anions and Cations

Qualitative analysis is the testing of a sample of matter to determine its composition. Qualitative analysis, applied by scientists over many years, revealed the composition of living things. In this experiment, you will become familiar with tests for qualitative analysis, the absence or presence of a compound, and how to standardize the tests. To demonstrate the process of qualitative testing you will test for several *anions* (negatively charged ions) and *cations* (positively charged ions) in solution. First you will test solutions of known composition to familiarize yourself with standardized positive results; then you will test a solution of unknown composition to determine which ions are present in it.

Qualitative analysis for ions is based on the fact that no two ions behave identically in all their chemical reactions. Each ion reacts in its own characteristic way. In this experiment you will identify the following anions: chloride, sulfate, carbonate and bicarbonate, and phosphate. You will also identify the following cations: iron, sodium, potassium, calcium, and ammonium.

Some of the identifying characteristics you should look for in doing the tests include (1) a color change in the solution, (2) the evolution of a gas, (3) the formation of a precipitate, and (4) a specific color generated in a flame test.

Chloride Ion Test. When a solution of silver nitrate is added to a solution containing chloride ions and nitric acid, a heavy bluish white precipitate of silver chloride forms. The equation for the reaction is:

$Ag^{+}(aq) + Cl^{-}(aq) \rightarrow AgCl(s)$ White precipitate

Sulfate Ion Test. A white precipitate of barium sulfate is formed in the presence of hydrochloric acid when a solution of barium chloride is added to a solution containing sulfate ions. The equation for the reaction is:

$Ba^{+2}(aq) + SO_4^{-2}(aq) \rightarrow BaSO_4(s)$ White precipitate

Carbonate and Bicarbonate Ion Test. The addition of hydrochloric acid to solutions containing carbonate or bicarbonate ions or to solid samples of carbonates and bicarbonates causes the rapid evolution (production) of carbon dioxide gas. The equations for the reaction of acid with these ions are:

$H^{+}(aq) + HCO_3^{-}(aq) \rightarrow H_2O(l) + CO_2(g)$

$2H^{+}(aq) + CO_3^{-2}(aq) \rightarrow H_2O(l) + CO_2(g)$

Phosphate Ion Test. When a solution containing phosphate ions is heated with a solution of ammonium molybdate [$(NH_4)_2MoO_4$] and dilute nitric acid a bright yellow precipitate of ammonium phosphomolybdate [$(NH_4)_3PO_4 \bullet 12MoO_4$] is formed. The yellow precipitate is extremely insoluble in nitric acid.

Ferric Ion Test. The blood-red color of the complex ion $Fe(SCN)^{+2}$ is obtained when a solution of potassium thiocyanate is added to an acidified solution containing ferric ions [iron (III) ions]. The equation for the reaction is:

$$Fe^{+3}(aq) + SCN^{-}(aq) \rightarrow [Fe(SCN)]^{+2}(aq) \qquad \text{Blood-red color}$$

Sodium Ion Test. Sodium ions are identified by the intense yellow color it produces in a flame test.

Potassium Ion Test. Potassium ions are identified by the violet color it produces in a flame test.

Calcium Ions Test. When a solution of sodium oxalate ($Na_2C_2O_4$) is added to a solution containing calcium ions, a white precipitate of calcium oxalate (CaC_2O_4) is formed. The equation for the reaction is:

$$Ca^{+2}(aq) + C_2O_4^{-2}(aq) \rightarrow CaC_2O_4(s) \qquad \text{White precipitate}$$

Ammonium Ion Test. When a strong base such as sodium hydroxide is added to a solution containing ammonium ions, the ammonium ions are converted to ammonia. Ammonia is easily detected by its odor. The equation for the reaction is:

$$NH_4^{+}(aq) + OH^{-}(aq) \rightarrow H_2O(l) + NH_3(g)$$

<u>I. Tests for Anions</u>

Hypothesis: No two anions behave identically in all their chemical reactions.

Objective: To identify anions in known solution using simple chemical tests. To use these qualitative tests to identify the anions in an unknown solution.

Materials and Equipment: 0.1 M Silver nitrate, 0.1 M sodium sulfate, 0.1 M barium chloride, 0.1 M sodium bicarbonate, 0.05 M sodium phosphate, 0.1 M ammonium molybdate, 0.1 M sodium chloride, 6 M nitric acid, 6 M hydrochloric acid, unknown solutions, high purity distilled water, small test tubes, test tube rack, 25mL graduated cylinder, 250mL beaker, dropper pipet, and water bath (ring stand, ring clamp, wire gauze, burner, 250mL beaker).

Procedure: Use only high purity water for rinsing glassware and diluting solutions in this experiment. Tap water contains ions that will interfere with your tests and give misleading information.

A. Test for Chloride Ion. Clean and rinse a test tube with high purity water. Add 2mL (about 20 drops) of sodium chloride solution to the test tube. Add about 2mL of dilute nitric acid to the tube and shake gently to mix the contents. Then add about 10 drops of silver nitrate solution to the tube. Shake to mix the solutions, and record your observations as the controlled positive in the data table.

B. Test for Sulfate Ion. Prepare a clean test tube as before. Add 2mL of sodium sulfate solution to the test tube. Add 2mL of 6 M hydrochloric acid to the tube and shake gently to mix the contents. Then add about 10 drops of barium chloride solution to the tube. Shake to mix the solutions, and record your observations as the controlled positive in the data table.

C. Test for Carbonate and Bicarbonate Ions. Prepare a clean test tube as before. Add 2mL of sodium bicarbonate solution to the test tube. Add 2mL of 6 M hydrochloric acid to the tube and shake gently to mix the contents. Shake to mix the solutions, and record your observations as the controlled positive in the data table.

D. Test for Phosphate Ion. Prepare a clean test tube as before. Add 2mL of sodium phosphate solution to the test tube. Add about 1mL of dilute nitric acid and 10 drops of 0.1 M ammonium molybdate solution to the tube. Shake gently to mix the contents. Place the tube in a boiling water bath for about 5 minutes. Remove the tube from the water bath, and then let it stand in a test tube rack for 10 minutes to cool. Record your observations as the controlled positive in the data table.

E. Test for Unknown. Repeat the four previous tests for anions with a solution that contains an unknown anion. Record your observations in the data table and compare your results with the known positives to determine which anion is in the unknown solution.

II. Tests for Cations

Hypothesis: No two anions behave identically in all their chemical reactions.

Objective: To identify anions in known solution using simple chemical tests. To use these qualitative tests to identify the anions in an unknown solution.

Materials and Equipment: 0.1 M iron (III) sulfate, 0.1 M sodium chloride, 0.1 M potassium thiocyanate, 0.1 M potassium chloride, 0.1 M calcium nitrate, 0.1 M sodium oxalate, 0.1 M ammonium nitrate, unknown solutions, dilute hydrochloric acid, dilute sulfuric acid, dilute sodium hydroxide, nichrome wire loops, cobalt-blue glass, small test tubes, test tube rack, red litmus paper, test tube holder, 100mL beaker, crucible tongs, and water bath.

Procedure: Use only high purity water for rinsing glassware and diluting solutions in this experiment. Tap water contains ions that will interfere with your tests and give misleading information.

A. Test for Ferric Ion. Clean and rinse a test tube with high purity water. Add 2mL of ferric sulfate solution to the test tube. Add 5 drops of dilute sulfuric acid and 5 drops of potassium thiocyanate solution to the tube and shake gently to mix the contents. Record your observations as the controlled positive in the data table.

B. Test for Sodium Ion. The presence of sodium is determined with a flame test. Prepare a clean test tube as before. To the test tube add 2mL of sodium chloride solution to the test tube. Add 3 drops of 6 M hydrochloric acid to the tube and shake gently to mix the contents.

Hold one end of a 10 cm length of nichrome wire by the handle and clean the nichrome wire by heating the loop end in a hot burner flame and dipping it, while it is still hot, into 6 *M* hydrochloric acid in a small beaker. Repeat this process several times until the flame remains almost colorless when the wire is inserted into the burner flame.

Dip the wire into the solution in the test tube, and then hold it in the hot burner flame. Record the result. After cleaning the wire thoroughly again, repeat the test. Since it is easy to contaminate test solutions with sodium ions, a faint yellow flame is not considered a positive test for sodium. Record your observations as the controlled positive in the data table.

C. Test for Potassium Ion. Like sodium, potassium is detected with a flame test. To a clean test tube add 2mL of potassium chloride solution. Add 3 drops of 6 *M* hydrochloric acid to the tube and shake to mix.

Clean your flame test wire, dip it into the test tube, and insert it in a hot burner flame. Record your results. If your solution contains sodium ions, the bright yellow sodium flame will obscure any violet color due to potassium. The bright yellow of the sodium flame can be filtered out by viewing the flame through a piece of cobalt-blue glass. Only the violet color of the potassium flame is visible through the cobalt-blue glass.

D. Test for Calcium Ion. Prepare a clean test tube as before. Add 2mL of calcium nitrate solution to the test tube. Add 10 drops of sodium oxalate solution to the tube, and warm the tube in a boiling water bath for a few minutes. Record your observations as the controlled positive in the data table.

E. Test for Ammonium Ion. Prepare a clean test tube as before. To the test tube add 2mL of ammonium nitrate. Add a few drops of dilute sodium hydroxide solution to the tube. Gently warm the contents of the tube while holding a piece of moistened red litmus paper at the mouth of the tube. Do not let the solution boil; otherwise, the sodium hydroxide will splatter the litmus paper and spoil the test. You also may smell the vapors coming out of the tube by cautiously wafting them towards you. Record your observations as the controlled positive in the data table.

Laboratory Report

Tests for Anions:

Solution	Observations	Conclusions
Sodium Chloride		
Sodium Sulfate		
Sodium Bicarbonate		
Sodium Phosphate		
Unknown		

The anions identified in the unknown are? _____

Tests for Cations:

Solution	Observations	Conclusions
Iron (III) Sulfate		
Sodium Chloride		
Potassium Chloride		
Calcium Nitrate		
Ammonium Nitrate		
Unknown		

The cations identified in the unknown are? _____

Post laboratory Questions

1. A test solution made with dilute nitric acid gives a white precipitate when treated with a few drops of silver nitrate. Name the anion present in the test solution.

2. You suspect a sample of high purity water is contaminated with sulfate ions. How would you test the water to confirm your suspicions?

3. How would you test a sample of urine for phosphate and chloride ions?

4. A water sample is contaminated with either sodium ions or sulfate ions. Describe the tests you would perform to identify the contaminating ions in the water sample.

5. How would you test a sample of urine for calcium ions?