

LAB: IDENTIFICATION OF ANIONS IN SOLUTION

- Purpose:**
- 1) To identify common anions in solution using simple qualitative chemical tests
 - 2) To use these tests to identify anions in an unknown solution

Intro:

The process of determining the composition of a sample of matter by conducting chemical tests is called **qualitative analysis**. In this experiment you will learn how to conduct qualitative tests to determine if certain **anions** (negatively charged particles) are present in solution. You will then use these tests to determine the ionic composition of an unknown solution.

Each ion is unique and reacts in its own, characteristic way. By conducting the appropriate tests and applying the rules of logic, the identities of the ions present in an unknown solution can be determined.

In this experiment, you will observe several types of chemical reactions that are commonly used as tests in qualitative analysis. These reactions include a color change, the evolution of a gas, and the formation of a **precipitate**. In precipitation reactions, two ionic solutions are combined and, upon mixing, a solid product called a precipitate is formed. This solid is a compound that "falls out of solution" because it is insoluble in water. A precipitate generally forms very quickly and in very fine particles. This gives the solution the appearance of cloudy water or thick paint, depending on the nature and the amount of the precipitate formed. The precipitate will slowly settle out of the solution. Testing for precipitate formation is done often to determine the ionic composition of solutions.

Materials:

AgNO ₃	silver nitrate
Na ₂ SO ₄	sodium sulfate
BaCl ₂	barium chloride
NaHCO ₃	sodium bicarbonate
(NH ₄) ₂ MoO ₄	ammonium molybdate
NaCl	sodium chloride
Na ₃ PO ₄	sodium phosphate
HNO ₃	nitric acid
HCl	hydrochloric acid
distilled water	
unknown solution	

8 clean, dry test tubes & rack
250 mL beaker & beaker tongs
ring stand & wire square
burner & striker
test tube tongs

Procedure:

NOTE: Volumes of solutions may be approximated in these tests. One milliliter is about 10 drops.

- 1) Prepare a boiling water bath for step #6. Fill a 250 mL beaker three-quarters full with tap water. Support the beaker on a wire square on the ring stand. Heat the water in the beaker to a SLOW boil using the gas burner.
- 2) The ion tests are very sensitive. Make sure your test tubes are clean and prepare them by rinsing them with a little distilled water. (Do not use tap water to prepare test tubes because it contains soluble ions which may give false positive test results.) Record the number of your unknown in your lab notebook.

3) **Test for CHLORIDE ion, Cl⁻**

Add approximately 2 mL of sodium chloride solution to a test tube. (This is the known Cl⁻). To a second test tube, add 2 mL of the unknown solution.

Add 2 mL of nitric acid to each test tube and gently flick the test tubes to mix the contents.
Caution: Nitric acid is corrosive.

Next, add 10 drops of silver nitrate solution to the first test tube which contains chloride ions. Gently flick the test tube to mix the contents and record any observations. Add 10 drops of silver nitrate to the unknown solution, mix, and record observations. Does your unknown contain chloride (Cl⁻) ions?

4) **Test for SULFATE ion, SO₄²⁻**

Add 2 mL of sodium sulfate solution to a clean test tube (This is the known SO₄²⁻). To a second test tube, add 2 mL of the unknown solution.

Add 2 mL of hydrochloric acid to each test tube and gently flick the test tubes to mix the contents.
Caution: Hydrochloric acid is corrosive.

Next, add 10 drops of barium chloride to the first test tube which contains sulfate ions. Flick gently to mix and record observations. Add 10 drops of barium chloride solution to the unknown solution, mix, and record observations. Does your unknown contain sulfate (SO₄²⁻) ions?

5) **Test for BICARBONATE ion, HCO₃⁻**

Add 2 mL of sodium bicarbonate solution to a clean test tube. (This is the known HCO₃⁻). To a second test tube, add 2 mL of the unknown solution.

Carefully observe each test tube as you add 2 mL of hydrochloric acid to each one. Is there any evidence of a chemical change? Record your observations. Does your unknown contain bicarbonate (HCO₃⁻) ions?

6) **Test for PHOSPHATE ion, PO₄³⁻**

Add 2 mL of sodium phosphate solution to a clean test tube. (This is the known PO₄³⁻). To a second test tube, add 2 mL of the unknown solution.

Add 1 mL of nitric acid and 1 mL of ammonium molybdate solution to the first test tube which contains phosphate ions. Gently flick to mix. Place the test tube in the hot water bath prepared in step #1. Heat the test tube for a few minutes and record any observations. Repeat the same steps for the unknown solution. Does your unknown contain phosphate (PO₄³⁻) ions?

TEACHER'S PREPARATION GUIDE

0.1 M	AgNO ₃	1.7g/100mL
0.1 M	Na ₂ SO ₄	1.4g/100mL
0.1 M	BaCl ₂	2.4g/100mL
0.1 M	NaHCO ₃	.84g/100mL
0.1 M	NaCl	.58g/100mL
0.05 M	Na ₃ PO ₄ 12H ₂ O	1.9g/100mL
6.0 M	HNO ₃	190 mL conc. HNO ₃ , into water to make 500 mL
6.0 M	HCl	250 mL conc. HCl, into water to make 500 mL
0.1 M	(NH ₄) ₂ MoO ₄	
	1)	Dissolve 40g of molybdic acid (NH ₄) ₆ Mo ₇ O ₂₄ 4H ₂ O in 58 mL of conc. NH ₃ (15 M) and 108 mL of water.
	2)	Slowly, with constant stirring, pour solution into a solution of nitric acid (196 mL conc. HNO ₃ and 460 mL water).
	3)	Keep mixture for several days.
	4)	OR : heat mixture to 40 °C until no more yellow precipitate. Decant solution from sediment. Store in glass stoppered bottles.

UNKNOWN S : (ANIONS)

# 1	=	HCO ₃ ⁻ , Cl ⁻
# 2	=	Cl ⁻ , PO ₄ ³⁻
# 3	=	PO ₄ ³⁻ , SO ₄ ²⁻
# 4	=	SO ₄ ²⁻ , Cl ⁻

Ion	Test Reagents	Test Results for Known Solution	Test Results for Unknown #	Ion Present in Unknown?
Cl^-				
SO_4^{2-}				
HCO_3^-				
PO_4^{3-}				

LAB: IDENTIFICATION OF CATIONS IN SOLUTION

Purpose: 1) To identify common cations in solution using simple qualitative chemical tests. 2) To use these tests to identify anions in an unknown solution.

Intro:

The process of determining the composition of a sample of matter by conducting chemical tests is called **qualitative analysis**. In this experiment you will learn how to conduct qualitative tests to determine if certain **cations** (positively charged particles) are present in solution. You will then use these tests to determine the ionic composition of an unknown solution.

Each ion is unique and reacts in its own, characteristic way. By conducting the appropriate tests and applying the rules of logic, the identities of the ions present in an unknown solution can be determined.

In this experiment, you will observe several types of chemical reactions that are commonly used as tests in qualitative analysis. These reactions include a color change, the evolution of a gas, and the formation of a **precipitate**. In precipitation reactions, two ionic solutions are combined and, upon mixing, a solid product called a precipitate is formed. This solid is a compound that "falls out of solution" because it is insoluble in water. A precipitate generally forms very quickly and in very fine particles. This gives the solution the appearance of cloudy water or thick paint, depending on the nature and the amount of the precipitate formed. The precipitate will slowly settle out of the solution. Testing for precipitate formation is done often to determine the ionic composition of solutions.

Materials:

$\text{Fe}_2(\text{SO}_4)_3$	iron (III) sulfate
NaCl	sodium chloride
KSCN	potassium thiocyanate
KCl	potassium chloride
$\text{Ca}(\text{NO}_3)_2$	calcium nitrate
$\text{Na}_2\text{C}_2\text{O}_4$	sodium oxalate
NH_4NO_3	ammonium nitrate
H_2SO_4	sulfuric acid
NaOH	sodium hydroxide

distilled water	forceps
unknown solution	test tube tongs
red litmus paper	burner & striker
wood splints & matches	ring stand & wire square
8 clean, dry test tubes & rack	250 mL beaker & beaker tongs

Procedure:

NOTE: Volumes of solutions may be approximated in these tests. One milliliter is about 10 drops.

- 1) Prepare a boiling water bath for step #6. Fill a 250 mL beaker three-quarters full with tap water. Support the beaker on a wire square on the ring stand. Heat the water in the beaker to a SLOW boil.
- 2) The ion tests are very sensitive. Make sure your test tubes are clean and prepare them by rinsing them with a little distilled water. (Do not use tap water to prepare test tubes because it contains soluble ions which may give false positive test results.) Record the number of your unknown in your lab notebook.
- 3) **Test for IRON (III) ion, Fe³⁺**
Add approximately 2 mL of iron (III) sulfate solution to a test tube. (This is the known Fe³⁺). To a second test tube, add 2 mL of the unknown solution. To the first test tube, add 5 drops of sulfuric acid and 5 drops of potassium thiocyanate. Flick gently to mix, then record observations. Repeat steps with the unknown solution. Does your unknown contain iron III (Fe³⁺) ions?
- 4) **Test for SODIUM ion, Na⁺**
Add 5 mL of sodium chloride solution to a clean test tube. (This is the known Na⁺). To a second test tube, add 5 mL of the unknown solution. Place a clean wood splint into each test tube and let them soak for a few minutes. After soaking, perform a flame test on the known solution. What color does sodium emit? Perform the flame test on the unknown solution. Does your unknown solution contain sodium (Na⁺) ions?
- 5) **Test for POTASSIUM ion, K⁺**
Add 5 mL of potassium chloride solution to a clean test tube. (This is the known K⁺). To a second test tube, add 5 mL of the unknown solution. Place a clean wood splint into each test tube and let them soak for a few minutes. After soaking, perform a flame test on the known solution. What color does potassium emit? Perform the flame test on the unknown solution. Does your unknown solution contain (K⁺) ions?
- 6) **Test for CALCIUM ion, Ca²⁺**
Add 2 mL of calcium nitrate solution to a clean test tube. (This is the known Ca²⁺). To a second test tube, add 2 mL of the unknown solution. To the first test tube, add 10 drops of sodium oxalate solution. Warm the test tube in the warm water bath for a few minutes. Record your observations. Repeat this test for the unknown. Does your unknown contain calcium (Ca²⁺) ions?
- 7) **Test for AMMONIUM ion, NH₄⁺**
Add 2 mL of ammonium nitrate to a clean test tube. (This is the known NH₄⁺).
To a second test tube, add 2 mL of the unknown solution.

To each test tube, add 3 drops of sodium hydroxide solution. **Caution: Sodium hydroxide is caustic.** Hold the test tube containing the ammonium nitrate solution with a test tube holder and GENTLY warm it along its sides with a back and forth motion through the burner flame. **DO NOT ALLOW THE SOLUTION TO BOIL.**

Moisten a piece of red litmus paper with distilled water. Hold the litmus paper near the mouth of the warm, test tube with the forceps. Cautiously sniff the vapors coming out of the test tube by wafting the vapors toward your nose. You should recognize the familiar odor. Record observations. Repeat the procedure on the unknown solution. Does your unknown contain ammonium (NH_4^+) ions?

TEACHER'S PREPARATION GUIDE CATIONS:

0.1 M	$\text{Fe}_2(\text{SO}_4)_3$	19.98 g/500 mL
0.1 M	NaCl	2.92 g / 500 mL
0.1 M	KSCN	4.86 g / 500 mL
0.1 M	KCl	3.74 g / 500 mL
0.1 M	$\text{Ca}(\text{NO}_3)_2$	8.20 g / 500 mL
0.1 M	$\text{Na}_2\text{C}_2\text{O}_4$	6.70 g/500 mL
0.1 M	NH_4NO_3	4.00 g / 500 mL
3.0 M	H_2SO_4	84 mL conc. H_2SO_4 , into water to make 500 mL
6.0 M	NaOH	120 g NaOH pellets into water to make 500 mL

UNKNOWN S : (CATIONS)

#1 = NH_4^+ , Ca^{2+}

#2 = NH_4^+ , Fe^{3+}

#3 = Ca^{2+} , Na^+

#4 = Ca^{2+} , Fe^{3+}

Ion	Test Reagents	Test Results for Known Solution	Test Results for Unknown #	Ion Present in Unknown?
Fe³⁺				
Na⁺				
K⁺				
Ca²⁺				
NH₄⁺				

LAB SUMMARY: IDENTIFICATION OF ANIONS & CATIONS IN SOLUTION

Student: _____

1. Define "precipitate".
2. List the names and formulas of the common laboratory acids used in these experiments.

3. Name these compounds:

NaOH _____	AgNO ₃ _____
Na ₃ PO ₄ _____	(NH ₄) ₂ MoO ₄ _____
KCl _____	NaHCO ₃ _____
KSCN _____	Na ₂ SO ₄ _____
NaCl _____	Fe ₂ (SO ₄) ₃ _____
BaCl ₂ _____	Na ₂ C ₂ O ₄ _____
Ca(NO ₃) ₂ _____	NH ₄ NO ₃ _____

4. Circle the ions that formed precipitates when tested:

Cl⁻ SO₄²⁻ HCO₃⁻ PO₄³⁻ Fe³⁺ Na⁺ K⁺ Ca²⁺ NH₄⁺

5. Circle the ions that formed gases when reacted:

Cl⁻ SO₄²⁻ HCO₃⁻ PO₄³⁻ Fe³⁺ Na⁺ K⁺ Ca²⁺ NH₄⁺

6. Circle the ions that could be identified by flame tests :

Cl⁻ SO₄²⁻ HCO₃⁻ PO₄³⁻ Fe³⁺ Na⁺ K⁺ Ca²⁺ NH₄⁺

7. Circle the anions in your unknown. (UNKNOWN # _____)

Cl⁻ SO₄²⁻ HCO₃⁻ PO₄³⁻ Fe³⁺ Na⁺ K⁺ Ca²⁺ NH₄⁺

8. Circle the cations in your unknown. (UNKNOWN # _____)

Cl⁻ SO₄²⁻ HCO₃⁻ PO₄³⁻ Fe³⁺ Na⁺ K⁺ Ca²⁺ NH₄⁺

CHAPTER 7 REVIEW*Chemical Formulas and Chemical Compounds***SECTION 7-1****SHORT ANSWER** Answer the following questions in the space provided.

1. _____ In a Stock name such as iron(III) sulfate, the roman numeral tells us _____.
- (a) how many atoms of Fe are in one formula unit
 - (b) how many sulfate ions can be attached to the iron atom
 - (c) the charge on each Fe ion
 - (d) the total positive charge of the formula unit
2. _____ The result of changing a subscript in a correctly written chemical formula is to _____.
- (a) change the number of moles represented by the formula
 - (b) change the charges on the other ions in the compound
 - (c) change the formula so that it no longer represents the compound it previously represented
 - (d) have no effect on the formula
3. The explosive TNT has the molecular formula $C_7H_5(NO_2)_3$.
- _____ a. How many elements make up this compound?
 - _____ b. How many oxygen atoms are present in one molecule of $C_7H_5(NO_2)_3$?
 - _____ c. How many atoms in total are present in one molecule of $C_7H_5(NO_2)_3$?
 - _____ d. How many atoms are present in a sample of 2×10^{23} molecules of $C_7H_5(NO_2)_3$?
4. How many atoms are present in each of these formula units?
- _____ a. $Ca(HCO_3)_2$
 - _____ b. $C_{12}H_{22}O_{11}$
 - _____ c. $Fe(ClO_2)_3$
 - _____ d. $Fe(ClO_3)_2$
5. _____ a. What is the formula for the compound dinitrogen pentoxide?
- _____ b. What is the Stock name for the covalent compound CS_2 ?
- _____ c. What is the formula for sulfurous acid?
- _____ d. What is the name for the acid H_3PO_4 ?

SECTION 7-1 continued

6. Some binary compounds are ionic, others are covalent. The types of bonding partially depend on the position of the elements in the periodic table. Label each of these claims as True or False; if False, specify the nature of the error.

a. Covalently bonded binary molecular compounds typically form from nonmetals.

b. Binary ionic compounds form from metals and nonmetals, typically from opposite sides of the periodic table.

c. Binary compounds involving metalloids are always ionic.

7. Refer to Table 7-2 on page 210 of the text and Table 7-5 on page 214 of the text for examples of names and formulas for polyatomic ions and acids.

a. Derive a generalization for when an acid name will end in the suffix *-ic* or *-ous*.

b. Derive a generalization for when an acid name will begin with the prefix *hydro-* and when it will not.

8. Fill in the blanks in the table below.

Compound name	Formula
Aluminum sulfide	
Aluminum sulfite	
	$PbCl_2$
	$(NH_4)_3PO_4$
Hydroiodic acid	