PURPOSE
To identify the ions in an unknown solution through the application of chemical tests.

BACKGROUND
Detectives in mystery novels often rush evidence from the crime scene to the lab for analysis. In this experiment, you will become a chemical detective. You will conduct laboratory analysis to determine the ionic composition of an unknown solution. The process of determining the composition of a sample of matter by conducting chemical tests is called qualitative analysis. Solutions of unknown ions can be subjected to chemical tests, and the results can be compared to the results given by known ions in the same tests. By conducting the appropriate tests and applying logic, the identities of the ions present in an unknown solution can be determined.

The analyses you perform are based upon the idea that no two ions produce the same set of chemical reactions. Each ion reacts in its own characteristic way. In this experiment, you will observe several types of chemical reactions commonly used as tests in qualitative analysis. These reactions include a color change, the production of a gas, and the formation of a precipitate—a solid product. As you do this experiment, remember that careful observation and logical reasoning are the keys to being a good detective. Who knows what ions lurk in your unknown solution?

MATERIALS (PER PAIR)
safety goggles and apron
250-mL beaker
gas burner
ring stand
wire gauze
crucible tongs
9 small test tubes
test-tube rack

25-mL graduated cylinder
dropper pipet
 nichrome wire (10-cm length)
cobalt-blue glass
test-tube holder
forceps
plastic wash bottle
distilled water
red litmus paper, 1 cm × 10 cm
0.1M ammonium molybdate
Dissolve 100 g of ammonium paramolybdate,
(NH₄)₆Mo₇O₂₄•4H₂O,
(or molybdic acid), in 145 mL of concentrated NH₃(aq) and
270 mL of water. Slowly, and
with constant stirring, pour
this solution into a solution
of nitric acid that has been
prepared by dissolving
490 mL of concentrated
HNO₃ in 1150 mL of water.
Keep the mixture in a warm
place for several days or
until a portion heated to
40°C deposits no yellow
precipitate. Decant the
solution from any sediment
and store in glass-stoppered
bottles.

6M hydrochloric acid
Pour 150 mL of concentrated
HCl slowly into 150 mL of
water.

6M nitric acid
Pour 190 mL of concentrated
HNO₃ slowly into 300 mL of
water and dilute to 500 mL.

6M sodium hydroxide
Dissolve 24 g NaOH pellets
in 80 mL of water, stirring;
cool. Dilute to 100 mL. Store
in a plastic bottle.

3M sulfuric acid
Pour 15 mL of concentrated
H₂SO₄ into 80 mL of water
and dilute to 100 mL.

red litmus paper
10-cm lengths

Place knowns and unknowns
in dropper bottles. Assign the
unknowns identification
codes. Unknowns may be
NaHCO₃, NaCl, Na₂PO₄,
Na₂SO₄, KCl, Fe₃(SO₄)₂,
NH₄NO₃, or Ca(NO₃)₂. Give
each pair a single unknown
to test along with the knowns
and identify at the end of the
procedure.

SAFETY FIRST!
In this lab, observe all precautions, especially the ones listed below. If
you see a safety icon beside a step in the Procedure, refer to the list below
for its meaning.

Caution: Wear your safety goggles. (All steps.)

Caution: Nitric acid, hydrochloric acid, sulfuric acid, and
sodium hydroxide are corrosive and can cause severe injury.
Never cover the opening of a test tube with your finger when
mixing chemicals in the tube. To mix the contents, “flick” the
tube as demonstrated by your teacher. (Steps 3, 4, 5, 6, 9, 10, 11,
12, 13.)

Caution: Silver, barium, and oxalate compounds are poisonous.
Avoid contact with these chemicals. (Steps 3, 4, 12.)

Caution: Silver nitrate will stain skin and clothing. (Step 3.)

Caution: Never pick up a dropper bottle by its cap. Always hold
a dropper with the tip lower than the rubber bulb so that the
liquid does not run into the bulb.

Caution: Exercise care when working with a hot water bath.
(Steps 1, 6, 12.)

Note: Return or dispose of all materials according to the
instructions of your teacher. (Steps 7, 14.)

Note: Wash your hands thoroughly after completing this
experiment.
PROCEDURE

As you perform the experiment, record your observations in Data Table 1.

Procedure note: In testing for different ions in Steps 3–13, you will always begin with two test tubes. For each step, you must add 2 mL of the known solution to one test tube and 2 mL of your unknown solution to a second test tube. The name of the known solution is given in each step. Note that 1 mL is approximately 20 drops. Count out 40 drops of water in a test tube and make note of the level of water in the tube. Throughout the experiment, fill a test tube to this same level whenever a 2-mL sample is called for. This practice will save you considerable time. Always clean the medicine dropper after each use.

Part A. Testing for Anions (Day 1)

1. Set up a boiling water bath for use in Step 6.

2. Thoroughly clean all the test tubes used in this experiment, rinsing them well with distilled water. Record the number of the unknown solution you will be testing.

3. Test for chloride ion, Cl \(^{-}\). Known solution is sodium chloride. CAUTION: Nitric acid is corrosive. Do not put your finger over the top of the tube to cover it. Add 2 mL of 6M nitric acid to each tube and gently flick the tubes to mix. Add 10 drops of silver nitrate to each tube and mix. Record your observations.

4. Test for sulfate ion, SO\(_{4}\)\(^{2-}\). Known solution is sodium sulfate. CAUTION: Hydrochloric acid is corrosive. Add 2 mL of 6M hydrochloric acid to each tube and mix. Add 10 drops of barium chloride solution to each tube and mix. Record your observations.

5. Test for hydrogen carbonate ion, HCO\(_{3}\)/H\(_{2}\)CO\(_{3}\). Known solution is sodium hydrogen carbonate. Carefully observe the test tubes as you add 2 mL of 6M hydrochloric acid to each tube. Record your observations.

6. Test for phosphate ion, PO\(_{4}\)\(^{3-}\). Known solution is sodium phosphate. Add 1 mL of 6M nitric acid and 10 drops of ammonium molybdate solution to each tube and mix. Place the tubes in a boiling water bath and heat for 5 minutes. Allow tubes to cool in a test-tube rack for 10 minutes. Record your observations. (Retain the water bath for Part B, Step 12.)

7. Follow your teacher’s instructions for proper disposal of the materials.

Part B. Testing For Cations (Day 2)


9. Test for iron(III) ion, Fe\(^{3+}\). Known solution is iron (III) sulfate. CAUTION: Sulfuric acid is corrosive. Add 5 drops of 3M sulfuric acid and 5 drops of potassium thiocyanate solution to each tube. Flick gently to mix. Record your observations.
Flame-test for sodium ion, Na⁺. Known solution is sodium chloride. Add 3 drops of 6 M hydrochloric acid to each tube. Flick gently to mix. Add 3–4 mL of 6 M hydrochloric acid to a small test tube in a test-tube rack. Heat the end of a 10-cm length of nichrome wire in a hot burner flame, as shown in Figure 16.1. While it is still hot, dip the end of the wire into the hydrochloric acid in the test tube. Remove the wire from the acid and immediately reheat it in the burner flame. Repeat this acid cleaning of the wire until the flame remains unchanged when the wire is heated.

Dip the acid-cleaned wire into the sodium chloride solution. Immediately hold it in the hot burner flame. Observe the color of the flame. Acid-clean the wire and then test the unknown solution. Record the color of the flame. (A faintly colored flame is not considered a positive test for sodium.) Save the prepared unknown solution for the next test.

11. Flame-test for potassium ion, K⁺. Known solution is potassium chloride. Add 3 drops of 6 M hydrochloric acid to each tube and mix gently. Acid-clean the nichrome wire. Flame-test the potassium chloride solution. Acid-clean the wire and test your unknown. If your unknown contains sodium, the color of the sodium flame will mask the color that is characteristic of potassium. You will be able to see the potassium color, if it is there, by looking at the flame through a piece of cobalt-blue glass. Record your results.

12. Test for calcium ion, Ca²⁺. Known solution is calcium nitrate. To each tube, add 10 drops of sodium oxalate solution. Warm the tubes in the boiling water bath for a few minutes. Record your observations.
Step 13.

\[ \text{NH}_4^+ + \text{OH}^- \rightarrow \text{NH}_3(aq) + \text{H}_2\text{O} \]

Gentle heating drives the NH\(_3\) from solution. The test paper is moistened so that, when the NH\(_3\) contacts it, the above reaction is reversed. The OH\(^-\) ions produced thereby cause the litmus to change from red to blue.

13. Test for ammonium ion, NH\(_4^+\). Known solution is ammonium nitrate. **CAUTION:** Sodium hydroxide can cause burns. To each tube, add 3 drops of 6M sodium hydroxide. Hold the tube containing the ammonium nitrate solution with a test-tube holder. Gently warm the tube along its sides using a back-and-forth motion through a burner flame. Do not allow the solution to boil. **CAUTION:** At all times, make sure that the opening of the tube is pointed away from other people. Hold a moistened piece of red litmus paper near the mouth of the test tube, as shown in Figure 16.2. The test will be spoiled if the solution contacts the litmus paper. Record the changes you observe. Fan the vapors coming out of the tube toward your nose with your hand. Cautiously sniff the vapors. Record your observations. Repeat the procedure for your unknown solution.

![Figure 16.2](image)

14. Follow your teacher's instructions for proper disposal of the materials.

Use the following disposal methods for chemical waste.

**Disposal 2:** NaCl(aq) in Step 10, and the materials in Step 12 (except materials required for optional flame test).

**Disposal 3:** Materials in Steps 5 and 6; HCl(aq) in Step 10; and the materials in Steps 9, 11, and 13. (Also, the materials from the optional flame test in Step 12.)

Because only drops of AgNO\(_3\) and BaCl\(_2\) are used in Steps 3 and 4, realistically, these materials can be flushed with water down the drain—**Disposal 2.** Alternatively, use **Disposal Method 5** for the materials in Step 4 and **Disposal Method 8** for the materials in Step 3.
### OBSERVATIONS

#### DATA TABLE 1: OBSERVATIONS

<table>
<thead>
<tr>
<th>Ion</th>
<th>Test Reagents/ Test Procedure</th>
<th>Test Results for Solution with Ion</th>
<th>Test Results for Unknown Solution No.___</th>
<th>Is Ion Present in Unknown?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl(^-)</td>
<td>H(_2)SO(_4), AgNO(_3)</td>
<td>white ppt.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SO(_4)(^{2-})</td>
<td>H(_2)SO(_4), BaCl(_2)</td>
<td>white ppt.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HCO(_3)(^-)</td>
<td>HCl</td>
<td>gas produced</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PO(_3)(^{4-})</td>
<td>HNO(_3), (NH(_4))(_2)MoO(_4)</td>
<td>canary-yellow ppt.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe(^{3+})</td>
<td>H(_2)SO(_4), KSCN</td>
<td>yellow solution turns deep red</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na(^+)</td>
<td>flame test</td>
<td>yellow flame</td>
<td></td>
<td></td>
</tr>
<tr>
<td>K(^+)</td>
<td>flame test</td>
<td>violet flame</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ca(^{2+})</td>
<td>Na(_2)C(_2)O(_4)</td>
<td>white ppt.; decomposes upon heating to produce another ppt. and a gas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NH(_4)(^+)</td>
<td>NaOH</td>
<td>gas produced upon heating; wet litmus paper turns blue when exposed to the gas</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### ANALYSES AND CONCLUSIONS

1. List the anions present in your unknown.
   
   **Answers will vary, depending upon makeup of unknowns.**

2. List the cations present in your unknown.
   
   **Answers will vary, depending upon makeup of unknowns.**
3. Write the complete ionic equation and the net ionic equation for the reactions that occur in Steps 3, 4, 5, 12, and 13.

\[ \text{Na}^+ (aq) + \text{Cl}^- (aq) + \text{Ag}^+ (aq) + \text{NO}_3^- (aq) \rightarrow \text{Na}^+ (aq) + \text{NO}_3^- (aq) + \text{AgCl}(s) \]

\[ \text{Ag}^+ (aq) + \text{Cl}^- (aq) \rightarrow \text{AgCl}(s) \]

\[ 2\text{Na}^+ (aq) + \text{SO}_4^{2-} (aq) + \text{Ba}^{2+} (aq) + 2\text{Cl}^- (aq) \rightarrow 2\text{Na}^+ (aq) + 2\text{Cl}^- (aq) + \text{BaSO}_4 (s) \]

\[ \text{Ba}^{2+} (aq) + \text{SO}_4^{2-} (aq) \rightarrow \text{BaSO}_4 (s) \]

\[ \text{Na}^+ (aq) + \text{HCO}_3^- (aq) + \text{H}^+ (aq) + \text{Cl}^- (aq) \rightarrow \text{H}_2\text{O}(l) + \text{CO}_2(g) + \text{Na}^+ (aq) + \text{Cl}^- (aq) \]

\[ \text{HCO}_3^- (aq) + \text{H}^+ (aq) \rightarrow \text{H}_2\text{O}(l) + \text{CO}_2(g) \]

\[ \text{Ca}^{2+} (aq) + \text{NO}_3^- (aq) + \text{Na}^+ (aq) + \text{C}_2\text{O}_4^{2-} (aq) \rightarrow \text{CaC}_2\text{O}_4(s) + \text{Na}^+ (aq) + \text{NO}_3^- (aq) \]

\[ \text{Ca}^{2+} (aq) + \text{C}_2\text{O}_4^{2-} (aq) \rightarrow \text{CaC}_2\text{O}_4(s) \]

\[ \text{NH}_4^+ (aq) + \text{NO}_3^- (aq) + \text{Na}^+ (aq) + \text{OH}^- (aq) \rightarrow \text{NH}_3(g) + \text{H}_2\text{O}(l) + \text{NO}_3^- (aq) + \text{Na}^+ (aq) \]

\[ \text{NH}_4^+ (aq) + \text{OH}^- (aq) \rightarrow \text{NH}_3(g) + \text{H}_2\text{O}(l) \]

4. It is possible to get a false-positive or a false-negative result when testing for ions. Propose a situation that could lead to a false positive for a particular ion. Choose a different ion and show how a false negative could result. Which do you think is more likely to happen—a false-positive or a false-negative result? Explain your reasoning.

A solution containing oxalate ions (but not chloride ions) could appear to test positive for chloride ions because silver oxalate is white and insoluble. A false negative could result because an ion is present in too low a concentration, from interference with another ion (for example, sodium ion interfering with the flame test for potassium), or from an improperly performed test. Both false-positive and false-negative results can occur, depending on the circumstances.
GOING FURTHER

Develop a Hypothesis

In the test for the sulfate ion (Step 4), hydrochloric acid was added before the barium chloride solution was added. Propose a hypothesis to explain why the hydrochloric acid is needed.

The hydrochloric acid is added to prevent the formation of a precipitate of barium hydroxide, which could produce a false-positive result.

Design an Experiment

Propose an experiment to test your hypothesis. If resources are available and you have your teacher’s permission, perform the experiment.

Test the hypothesis by adding barium chloride to aqueous solutions over a range of pH, and determine the approximate pH at which barium hydroxide will precipitate.