Testing for Cations and Anions
Revised Procedure

Lab Information

Comments: Use several small test tubes to hold the reagents. Be sure to label each test tube. HCl and HNO₃ are strong acids and NaOH is a strong base. Handle with care! If they are spilled on the skin, rinse thoroughly with water for 10 minutes.
- Dispose all waste in the Chem 101 Waste Jar...
- Tear out the report sheets and place them beside the matching procedures.
- Related Topics: Ions, chemical change, solubility rules

Experimental Procedures
WEAR YOUR GOGGLES!

A. Tests for Positive Ions (Cations)

Materials: Bunsen burner, test tubes and test tube rack, flame-test wire, dropper bottles of 0.1 M NaCl, 0.1 M KCl, 0.1 M CaCl₂, 0.1 M (NH₄)₂C₂O₄ (ammonium oxalate), 0.1 M NH₄Cl, 0.1 M FeCl₃, 0.1 M KSCN (potassium thiocyanate), 3 M HCl, 6 M HNO₃, 6 M NaOH, red litmus paper, warm water bath, stirring rod

USE OF THE LABORATORY BURNER

NOTE: Before attempting to light a Bunsen burner, make sure that you are successful in generating sparks out of the striker. If you are not successful in getting the burner lit after two attempts, TURN OFF THE GAS FROM THE GAS JET.

Bunsen burners are located in the metal ware drawer. Always check the rubber tubing for holes. Most heating in your chemistry course is done with a gas burner. In this laboratory you will use a burner of the Bunsen Type. The Bunsen burner has a gas needle valve at the base of the burner to control the amount of gas and in that way, the size of the flame. The burner also has an air inlet just above the gas inlet, which can be adjusted by screwing or unscrewing the barrel of the burner. This adjustment determines the amount of air mixes with the gas - the larger the air opening, the hotter the flame.

The fuel used for the burner is natural gas. You will find a natural gas jet at each work area. Always be sure the gas jet is shut off completely when the burner is not lit.

To light the burner:
- Close the gas needle valve of the burner (turn counter clockwise).
- Adjust the barrel of the burner so that you see an air opening.
- Turn the gas jet 90 degree.
- Adjust the gas needle valve of the burner clockwise until you hear the gas.
- Light the burner with a striker. Adjust the air control to get a blue, nearly transparent flame.

If the air inlet is closed and the gas is lit, the flame will be large and luminous. The light is the radiation given off by the hot carbon particles that are burned only partially. This luminous flame is not very hot and dangerously flimsy. This very cool flame type will never be used in this lab. If the air control is adjusted so that air is mixed with the gas before it gets to the flame, the flame will become less luminous, and finally blue. When the air is adjusted correctly to give the hottest flame,
it will look something as shown in the picture. The inner cone of the flame is pale blue, and the outer cone is pale violet. The inner cone contains the unburned gas that is hot enough to radiate light. The hottest point is just above the inner cone.

Testing for Cations and Anions

Preparation of an unknown

Get your unknown from the stockroom. Transfer the sample label to your data sheet. Use small portions of this unknown solution in each of the tests. You will need to identify the presence of a cation and an anion in the unknown by comparing the test results of the unknown with the test results given by each of the known solutions.

Part A: General Notes

- All reagents are in dropper bottles and complete sets of reagents are setup on each work island.
- You will add the reagents directly from the dropper bottles into your test tubes. There are 40 drops in two mL of solution.
- Your unknown will only have 1 cation and 1 anion.
- A community water bath will be set up to 60°C in the Southwest hood.

A.1 Flame tests for Na⁺, K⁺, and Ca²⁺

The sodium flame was done as demo in lecture. Do not perform a flame test on solution Na²⁺ -it takes 45 minutes to burn off!

- Obtain 4 test tubes and place 5-8 drops of 0.1 M solutions of KCl, CaCl₂, 3 M HCl, and your unknown solution into separate test tubes.
- Check out a wire loop. Light a Bunsen burner and adjust to a hot flame. Clean the wire loop by dipping the test wire in 3 M HC1 and heat the wire on the hottest part of the flame until the flame is light blue.
- Place the wire loop in the KCl solution and then into the flame.
- Record the color produced by the K⁺ ion. The color (pink-lavender) of K⁺ does not last long, so look for it immediately.
- Clean the wire, and repeat the test with the CaCl₂ solution. Record the color of the Ca²⁺ flame.

Testing the unknown

- Clean the flame-test wire and dip it in the unknown solution. Record the results when the wire is placed in a flame. If there is a color that matches the color of an ion you tested in the flame tests (A.1), you can conclude that you have one of the ions Na⁺, K⁺, or Ca²⁺ in your unknown. Record your observation.
- If you think Ca²⁺ is present, you may wish to confirm it with test A.2. If the flame test does not produce any color, then Na⁺, K⁺, and Ca²⁺ are not present in your unknown.

A.2 Test for calcium ion, Ca²⁺

- Place 2 mL of 0.1 M CaCl₂ in a test tube and 2 mL of your unknown solution in another test tube.
• Add 15 drops of ammonium oxalate solution, 0.1 M (NH₄)₂C₂O₄, to each. Look for a cloudy, white solid (precipitate).

• If the solution remains clear, place the test tube in a warm water bath for 5 minutes and look for a precipitate. The net equation for the reaction is

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

A while precipitate indicates the presence of Ca²⁺.

A.3 Test for ammonium ion, NH₄⁺

• Moisten a strip of red litmus paper. Place 2 mL of 0.1 M NH₄Cl in a test tube and 2 mL of your unknown in another test tube.

• Add 15 drops of 6 M NaOH to each. Carefully fan the vapors from the test tube toward you. You may notice the odor of ammonia.

• Place the strip of moistened red litmus paper across the top of the test tube and set the test tube in a warm water bath. The NH₃ (g) given off will turn the red litmus paper blue.

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

Ammonia

• Repeat the test with your unknown. Record the results.

A.4 Test for ferric ion, Fe³⁺

• Place 2 mL of 0.1 M FeCl₃ in a test tube and 2 mL of your unknown in another test tube.

• Add 5 drops of 6 M HNO₃ and 2-3 drops of potassium thiocyanate, 0.1 M KSCN. A deep red color indicates that Fe³⁺ is present. A faint pink color is not a positive test for iron. Repeat the test with your unknown. Record the results.

\[
\text{Fe}^{3+} + 3\text{SCN}^- \rightarrow \text{Fe(SCN)}_3
\]

Deep red color

Tests for Negative ions (Anions)

Materials: Test tubes, test tube rack, 0.1 M NaCl, 0.1 M AgNO₃ (dropper bottle), 3 M HCl, 6 M HNO₃, stirring rod, 0.1 M Na₂SO₄, 0.1 M BaCl₂, 0.1 M Na₃PO₄, (NH₄)₂MoO₄ (ammonium molybdate reagent), 0.1 M Na₂CO₃, hot water bath

B.1 Test for chloride ion, Cl⁻

• Place 2 mL of 0.1 M NaCl solution in a test tube and 2 mL of your unknown in another test tube.

• To each sample, add 5-10 drops of 0.1 M AgNO₃ and 10 drops of 6 M HNO₃. Stir with a glass stirring rod. Caution: AgNO₃ stains the skin. Any white solid that remains is AgCl(s). Any white solids that dissolve with HNO₃ do not contain Cl⁻. Record the results of your known and unknown.
White precipitate remains after HNO₃ is added.

B.2 Test for sulfate ion, SO₄²⁻

- Place 2 mL of 0.1 M Na₂SO₄ solution in a test tube and 2 mL of your unknown in another test tube.
- Add 1 mL (20 drops) of BaCl₂ and 5-6 drops of 6 M HNO₃ to each test tube. BaSO₄, a white precipitate, does not dissolve in HNO₃. Other anions, CO₃²⁻ and PO₄³⁻, will also form barium compounds, Ba₃(PO₄)₂ and BaCO₃, but they will dissolve in HNO₃.
- Record your test results for the known and unknown.

B.3 Test for phosphate ion, PO₄³⁻

Place 2 mL of 0.1 M Na₃PO₄ solution in a test tube and 2 mL of your unknown in another test tube. Add 10 drops of 6 M HNO₃ to each. After the test tubes are warmed in a hot water bath (60°C), add 5 drops of ammonium molybdate solution, (NH₄)₂MoO₄. The formation of a yellow precipitate indicates the presence of PO₄³⁻. Record the test results of the known and the unknown.

B.4 Test for carbonate ion, CO₃²⁻

- Place 2 mL of 0.1 M Na₂CO₃ solution in a test tube and 2 mL of your unknown in another test tube.
- While carefully observing the solution, add 10 drops of 3 M HCl to each sample. Watch for a strong evolution of bubbles of CO₂ gas as you add the HCl. The gas bubbles are formed quickly, and may be overlooked. If gas bubbles were not observed, add another 15-20 drops of HCl as you watch the solution.

Na₂CO₃(s) + 2HCl(aq) ———> CO₂(g) + H₂O + 2NaCl(aq)
Gas bubbles
- Record your results for the known and the unknown.

C. Writing the Formula of Your Unknown Salt

Your unknown solution was made from a salt composed of a cation and an anion. From your test results, you can identify one of the cations (Na⁺, K⁺, Ca²⁺, NH₄⁺, or Fe³⁺) and one of the anions (Cl⁻, SO₄²⁻, PO₄³⁻, or CO₃²⁻). For example, if you found that in the cation tests you got the same test result as for Ca²⁺ and in the anion tests you got the same result as for Cl⁻, then the ions in your unknown salt would be Ca²⁺ and Cl⁻. The formula CaCl₂ is written using charge balance.

C. 1 Write the symbols and names of the cation and anion that were present in your unknown.

C.2 Use the ionic charges of the cation and anion to write the formula and name of the salt that was your unknown.

Skip Part D (Open Lab)