

Object: To identify specific nonmetal anions contained in solution, (2) to use a flame test for metal cations, (3) to identify the ions present in an unknown sample.

Background: One of the major fields of chemistry is analytical chemistry. Analytical chemistry is made up of two main areas: quantitative analysis (to determine the concentration of a substance) and qualitative analysis (to determine which elements or compounds are present). In this lab we will qualitatively examine a number of substances to determine which ions are present in a sample.

One technique we employ is analysis of the light produced from an atom. This technique falls under an area of chemistry called spectroscopy. Spectroscopy can be used in many areas for detection of atoms present such as determining the chemical makeup of stars or analyzing water for different substances.

*Colors of light:* Sodium chloride is an ionic substance that is made up of a cation (positive charge) and an anion (negative charge). A method of determining which cation is in a compound is by a flame test. When energy is added (such as when the cation-containing substance is placed in a flame) each cation produces a particular color due to its light emission spectrum. The unique yellow-orange color of a sodium lamp is due to this phenomenon. In this lab, a wire tool called a flame loop will be used to hold a thin layer of solution when it is passed through a flame. The color of the flame will indicate which cation is present.

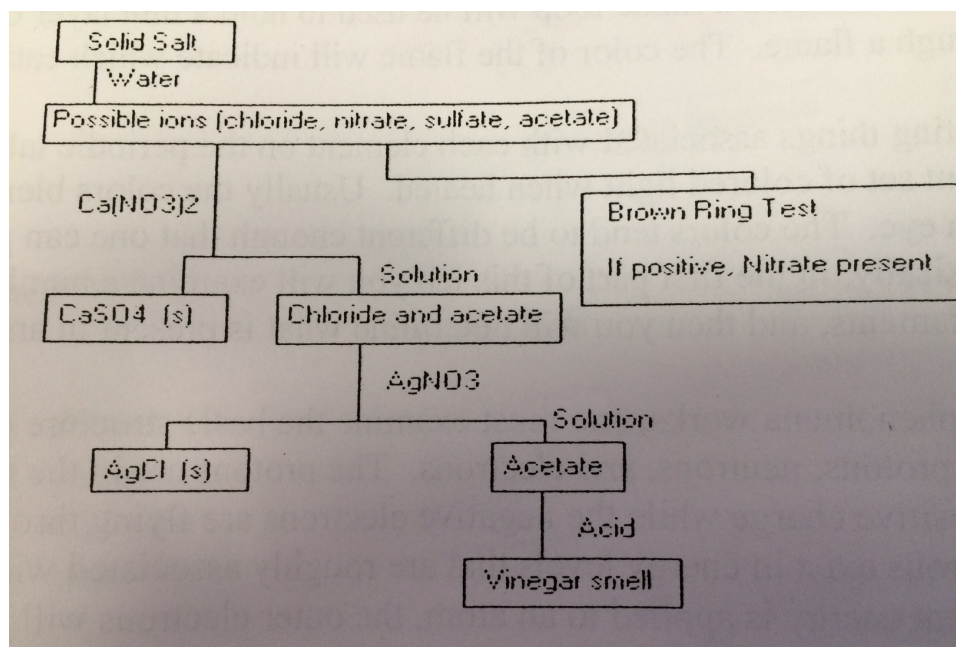
One of the most interesting things associated with each element on the periodic table is that each will give off a different set of colored light when heated. Usually the colors blend and produce one perceived color to the human eye. The colors tend to be different enough that one can pick out specific atoms in a sample. Basically, in the first part of this lab you will examine a number of colors associated with known elements, and then you will determine what is present in an unknown sample.

To understand how this phenomenon works, one must examine the basic structure of atoms. Atoms are composed of protons, neutrons, and electrons. The protons and neutrons are in the central nucleus while the lighter electrons fly around the nucleus. Electrons exist in energy levels that are roughly associated with their distance from the nucleus. As energy is added to an atom, the outer electrons jump to higher energy levels. When the atom is removed from the heat, the electrons return to their original levels. In the process, the excess energy absorbed by jumping to higher levels is given off as light. Because each kind of atom has a different number of protons and electrons, the attraction of any single electron to the nucleus will vary from element to element. It therefore takes different amounts of energy to cause an electron to jump in different kinds of atoms. Because different amounts of energy are absorbed, when the electrons jump back down they give off different amounts of light energy, which we see as different wavelengths or colors of light. So each element produces a different color pattern and allows for detection.

*Detection of Four Anions:* The presence of a particular anion can be determined by reactions unique to that anion. Often this determination involves a scheme, or series of reactions. When the ions of normally insoluble ionic compounds are placed in the same solution, they form a solid precipitate. Many times a precipitation reaction can determine what ion or ions are present in a solution.

Four main anions will be involved in this experiment, namely chloride ( $\text{Cl}^-$ ), sulfate ( $\text{SO}_4^{2-}$ ), nitrate ( $\text{NO}_3^-$ ), and acetate ( $\text{C}_2\text{H}_3\text{O}_2^-$ ). All the unknown salts will be soluble in water. The original solution will first be tested for nitrate by what is known as the brown ring test. The original solution remains in the iron sulfate solution. The nitrogen in nitrate at the interface is reduced by the sulfuric acid forming the complex nitrosyliron (II) ion ( $\text{Fe}(\text{NO})^{2+}$ ), which is a brown color. If no brown ring forms, nitrate is not present in the solution and other tests will be performed to find the correct anion. When the calcium ion is added to a solution only the sulfate ion will produce a precipitate. If there is no precipitate, then no sulfate ion is present. The solution left will precipitate only the chloride ion when the silver ion is added. If a precipitate still does not form, the remaining solution can be tested for the acetate ion by adding an acid. The acetate ion plus a hydrogen ion generates acetic acid that smells like vinegar.

The sequence of reactions involved in a qualitative analysis is sometimes referred to as a “qual scheme.” A chemist would write a representation of this sequence using vertical lines to represent a separation of the solution into solid products and the remaining ions.



*Waste Treatment:* All original test solutions can be flushed down the drain with plenty of water except the barium solution. Barium is classified as a toxic chemical. The barium should be precipitated with sulfuric acid. The solid barium sulfate can be filtered out and buried in a toxic waste dump. The calcium sulfate, silver chloride, and silver nitrate precipitates can be placed in the trash. Concentrated acids can cause damage to the plumbing if they are not diluted and/or neutralized before disposal.

### Procedure:

#### *Cation Test:*

1. Obtain the bottles containing the 5 known cation test solutions and the two unknowns.
2. Light a bunsen burner. Adjust it to a blue flame. (Be careful; don't burn yourself.)
3. Place a few drops of one of the solutions on the tip of a cotton swab. Place the tip in the flame and record the color produced. Watch closely because the cotton may produce a small orange color. If it is faint, add drops of the solution and try multiple times.
4. Repeat the process for each solution, using a clean cotton swab. Record the color of each in the Data section.
5. Based on the colors produced from the known solutions, perform the same test on the unknown cations. Record the identity of the unknowns in the Data section.

#### *Anion Test:*

1. Place 0.25 g (a scoop  $\frac{1}{2}$  the size of a pea) of the unknown powder in a small beaker and 30 mL of distilled water. This will be your stock solution.
2. Pour about 5 mL of the stock solution into a test tube.
3. Perform the nitrate brown ring test by adding 5 drops of iron (II) sulfate ( $\text{FeSO}_4$ ) to the test tube. Mix well by swirling. Hold the test tube at about a 45 degree angle. Carefully drop 5 drops of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ) down the side of the test tube. The liquids should form two layers with the sulfuric acid on the bottom. Set the test tube aside in an upright position for a few minutes and continue with the other tests. When finished, look to see if a brown ring forms at the interface between the two layers. If so, this is a positive test for the nitrate ion.
4. Pour about 5 mL of the stock solution into a new test tube for the Sulfate Test. Add 5 drops of 1 M calcium Nitrate ( $\text{Ca}(\text{NO}_3)_2$ ). If a precipitate occurs, this is a positive test for the anion present being sulfate.
5. If there is no precipitate, add 2 drops of 1 M silver nitrate ( $\text{AgNO}_3$ ) to the solution. If there is a precipitate, the anion is chloride.  

Note:

 Silver nitrate on skin causes a brown stain that will not go away for about three days.
6. If there is no precipitate, add 2 drops of 1 M HCl. Silver nitrate will precipitate. Waft or fan the fumes from the test tube to your nose. A vinegar smell is a positive test for acetate ion. Don't let the white precipitate fool you. You are only looking for a vinegar smell.
7. Record the identity of the anion on the data sheet.
8. Use some fresh stock to perform a flame test to determine the cation in the solution. You should now be able to determine the compound's identity. Record this on the data sheet.
9. Repeat the Anion Test experiment on a second unknown.

Data:

*Liquid Cation Samples*

Flame Test Color

Sodium	_____
Potassium	_____
Lithium	_____
Copper	_____
Barium	_____
Unknown 1	_____
Unknown 2	_____

*Solid Unknown Samples*

*Anion Test*

	POSITIVE	NEGATIVE
Nitrate test	_____	_____
Sulfate test	_____	_____
Chloride test	_____	_____
Acetate test	_____	_____

Sample number \_\_\_\_\_

SALT

CATION

ANION

Chemical formula: \_\_\_\_\_

	POSITIVE	NEGATIVE
Nitrate test	_____	_____
Sulfate test	_____	_____
Chloride test	_____	_____
Acetate test	_____	_____

Sample number \_\_\_\_\_

SALT

CATION

ANION

Chemical formula: \_\_\_\_\_

Discussion Questions:

1. What is analytical chemistry?
2. Why do atoms give off light when heated?
3. Why are the colors produced different for each type of atom?
4. How can one use this lab to solve problems in science, or in life?

Conclusions: