7 Qualitative Analysis II: Cations II

Version: March 11, 2004

- **Gain additional experience in the identification of cations in solution.**
- **Use flame tests to identify certain cations.**
- **Learn some of the descriptive chemistry of some common cations.**

DISCUSSION

In Experiment 6 you saw how acid-base, solubility, and complex ion equilibria could be used to separate and identify cations. However, some cations do not form any common insoluble compounds or complex ions that can be used in such a separation scheme. In this experiment you will learn other techniques for identifying such cations.

One such technique involves the emission of light from excited atoms in a flame. Recall from your study of atomic structure that electrons in an atom can absorb energy and enter a higher energy level. The atom is then in an ***excited state*****, possessing more energy than its normal or *ground state*.** One way the atom can rid itself of this excess energy and return to the ground state is to emit electromagnetic radiation (often in the form of visible light).

Each element has its own individual emission spectrum, a "fingerprint" by which it can be identified. When the emitted light is in the visible region, we see a color that is characteristic of that element. The presence or absence of a certain color often can be used to determine the presence or absence of an element in a sample.

If you have seen a fireworks display, you have seen the characteristic colors of several elements. When a fireworks shell explodes, combustion reactions producing very high temperatures cause excitation of atoms, followed by the emission of colored light. Commonly seen are reds from strontium, greens and blues from barium and copper, yellows from sodium, and whites from magnesium or aluminum.

When placed in the flame of a bunsen burner, many elements undergo excitation and emission similar to that which occurs in a fireworks display. You will use this flame emission technique to identify sodium and potassium. The sodium flame color is bright yellow and almost impossible to miss, but the pale violet color of potassium in a flame often is difficult to detect even for trained observers. If the flame is viewed through two thicknesses of cobalt glass, which filters out all colors except violet, results are improved. The violet potassium flame appears reddish when viewed through the glass. With practice you will be able to detect potassium even when sodium is present.

The ammonium ion, which does not emit visible light when excited, will be detected by its conversion to ammonia in a strongly basic solution. Ammonia is volatile and can be detected above a solution by using moistened litmus paper.

PROCEDURE

1. Preliminary Observations

***a*. Flame tests.** Obtain a piece of nichrome wire with a small loop in one end. On the other end is fastened a handle made of a piece of glass tubing or a cork. Clean the wire by dipping it into a small amount of concentrated HCl*(aq)* in a small test tube, then holding it in the flame of a bunsen burner just above the tip of the inner bright blue cone. Repeat this process until the wire no longer colors the flame after a few seconds. Next dip the wire into some 0.1 *M* KCl*(aq)* (or KNO_3) and hold it in the flame. Look carefully for the violet color of potassium. Repeat the test, this time viewing the flame through two thicknesses of cobalt glass. The color will be visible only briefly. If you have difficulty seeing the color, either concentrate some of the solution in an evaporating dish, or do the test in a dark room, or both. Continue testing until you have some confidence in your ability to see the potassium color. Record the color on the report.

Repeat the above procedure with a 0.1 *M* NaCl solution (after cleaning the wire with HCl). Notice the bright, sustained color of sodium. Notice also that it is not visible when viewed through the cobalt glass. Record the color of the sodium flame on the report.

Now mix together some NaCl*(aq)* and some KCl*(aq)*, and test as before. Notice that the yellow sodium flame completely obscures the potassium flame unless the cobalt glass is used. Continue working with these known solutions until you are confident in your ability to detect sodium and potassium in a solution.

***b*. The test for NH_4^+.** To 2 mL of 0.1 *M* NH_4Cl*(aq)* in a small test tube, add a drop or two of 6 *M* NaOH*(aq)*, making sure the base does not run down the side of the tube. Mix the solution well, then hold a piece of moistened red litmus paper in the mouth of the test tube. It should start to turn blue within a few seconds. Warming the test tube slightly will speed up the test. Try this again with a more dilute NH_4Cl solution, with distilled water, and with 0.1 *M* NaCl*(aq)* so you will be familiar with the appearance of a positive and a negative test. If the litmus paper contacts the strongly basic solution, it will of course turn blue and invalidate the test.

2. Analysis of the Unknowns

As in the last experiment, exchange two of your clean test tubes for two unknowns at the stockroom window. These unknowns will contain one or more of the cations Na^+, K^+, and NH_4^+. Mix each unknown thoroughly and record the unknown numbers on the report sheet. Proceed as outlined below.

a. Do a flame test on a small sample of each unknown, using the cobalt glass and concentrating the solution if necessary. Record your observations on the report.

b. Test for NH_4^+ as described in the preliminary observations above, using a new 2 mL sample of unknown. Record your observations.

3. Confirming the Analysis

If time permits and to be more certain of your results, make solutions matching the suspected composition of your unknowns by mixing the reagents available on the shelves. (Use chlorides or nitrates). Test these solutions, comparing the results with the observations you made on your unknowns.

7 Qual Analysis II: Cations II

Name ______________________________
Section __________________Locker ____
Instructor ___________________________

1. Preliminary Observations

Flame Tests. Describe the colors you observed when solutions containing the following ions were placed in a flame.

Ions	Without cobalt glass	With cobalt glass
K^+		
Na^+		
K^+ and Na^+		

Why is the sodium flame not visible through the cobalt glass?

Explain the origin of the light produced when solutions containing sodium or potassium are placed in a flame.

Why are different colors of light produced by sodium and potassium?

b. **The Test for NH_4^+**. Write the equation for the reaction that occurs when $NaOH_{(aq)}$ is added to a solution containing NH_4^+.

2. Analysis of the Unknowns

Complete the following table for your unknowns:

Unknown	Step	Observations	Conclusion

Summarize your analysis in the following table:

Unknown Number	**Ions Found**

3. Confirming the Analysis

Describe below what you did to confirm that your analysis is correct.

APPLICATION OF PRINCIPLES

1. Barium and calcium emit green and orange light, respectively, in a flame test. Neither color is bright enough to interfere with the sodium test, but both may interfere with the potassium test even when cobalt glass is used. How would you test for potassium in an unknown that contained barium or calcium?