Experiment 8b
Quantitative Analysis of Water

INTRODUCTION

In Part B, you will determine if certain ions are present in the water by chemically reacting these ions with another substance to form a new compound (called selective precipitation. One way to detect the presence of a substance in water is to perform a precipitation reaction where selected ions undergo a chemical change to form new specific products. When the presence of a substance is desired and not the amount of the substance, the method of analysis is called qualitative. The analysis of drinking water is used to identify the substances which are present in the homogeneous mixture and to detect certain toxic chemicals.

One toxic component of drinking water is lead (Pb). Lead is classified as a B2 carcinogen, it is known to have cancer-causing properties in animals but there is insufficient data on its effect on humans. A major source of lead in drinking water is the lead plumbing materials and fixtures. One of the problems with lead (Pb^{2+}) depends on its suspected ability to replace calcium (Ca^{2+}) in bones and tissue. You will be able to test for lead in your water sample but be aware that this type of analysis will only detect fairly large quantities of the substance. The maximum contaminant level for lead is 0.06 ppm therefore, hopefully, its presence will not be detected. Other substances which you will analyze for include the chloride ion (Cl^-), sulfate polyatomic ion (SO_4^{2-}), and both calcium and magnesium ions (Ca^{2+} and Mg^{2+}). The latter two ions can not be selectively precipitated from each other by the described method. These ions were chosen because the average contaminant level is 500 ppm each for the first two and 180 ppm combined for the last two. For more information about the drinking water in your area contact the water department in your local city government.

PROCEDURE

i. In a 250 mL beaker, add about 125 to 150 mL of water (tap or sea water) and 3 boiling stones.

ii. Gently boil this solution down to about 15 to 20 mL for tap, down to 40 to50 for sea water. As the solution is boiling, label 9 clean 100 mm (smallest) test tubes (#1 to #9). When the water level reaches about 50 to 75 mL solids (a white residue on the sides of the beaker) may appear. This is an undesirable effect so two actions must be taken. If solids begin to appear as the water is boiling then (1) extract about 5 to 10 mL of the concentrated water into a clean 50-mL beaker then (2) add about 3 drops of dilute nitric acid (HNO_3) to

the water in the 250 mL beaker. The acid will re-dissolve the solids back into the water. Stir well after adding the nitric acid. If the solids remain, add a couple more drops of acid, the solution should be slightly acidic to avoid precipitation of hydroxides and other compounds. Remove the flame. Allow the concentrated solution to cool to room temperature.

To test tubes #6 through #9, add approximately 2 mL (about 40 drops) of each of the following solutions. These are your known samples. You will compare the expected known reactions to those observed with your water sample.

#6 = 0.2 M sodium chloride
#7 = 3.0 M sulfuric acid
#8 = 0.05 M lead(II)nitrate
#9 = 0.2 M calcium nitrate or magnesium nitrate

iii. Divide the approximately 15 mL concentrated water solution into the 4 separate 100 mm test tubes pre-labeled as #1 through #5. The anions will be analyzed for first. Test tube #1 will be used to test for the chloride ion (Cl^-), and #2 is for sulfate (SO_4^{2-}). Next you will test for cations, test tube #3 will be used to test for lead (Pb), and #4 will be used to test for calcium/magnesium. Test tube #5 is for the sodium test.

iv. Test for each anion and cation using the following procedures. The anions will be first then the cations.

I. THE TEST FOR CHLORIDE

A. To test tube #1, add about 4 drops of dilute nitric acid to make sure the solution is acidic. Test with litmus paper. Result: _____

B. To test tube #6, add 5 drops of 0.1 M silver nitrate ($AgNO_3$). Does a precipitate form? What color is it? Add 5 more drops if a solid does not appear. Repeat this test with test tube #1. Does a precipitate form? **Yes** or **No**? (Circle one)

C. Write the molecular equation which describes the reaction in test tube #6. In your water, the silver ions (Ag^+) from silver nitrate react with the chloride ions in your water sample to form a precipitate silver chloride (AgCl). Note that the atoms were rearranged to form a new substance. This is a chemical change. Write the net ionic equation for the reaction of silver ions with chloride ions for Question 3 on the report page (page 4).

II. THE TEST FOR SULFATE

A. To test tube #7, add 5 drops of 0.1M barium nitrate ($Ba(NO_3)_2$). Does a precipitate form? What color is it? Add 5 more drops if a solid does not appear. Repeat this test with test tube #2 (water). Record your observations here.

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B. Write the molecular equation which describes the reaction in test tube #7 for Question 3 on the report page (page 4). In your water sample, the barium ions (Ba^{2+}) from barium nitrate react with the sulfate ion in your water solution to form the new substance barium sulfate ($BaSO_4$). Write the net ionic equation for the reaction of barium ions with sulfate ions for Question 3 on the report page (page 4).

III. THE TEST FOR LEAD

A. To test tube #8, add 4 drops of 6M HCl (hydrochloric acid). Again if a solid does not appear add 4 more drops. What color is the solid formed? Write a balanced molecular equation for Question 3 on the report page (page 4). Repeat this test with your water sample (test tube #3). If a solid does not appear by 10 drops then the amount of lead in your sample is too small to be detected by this method of analysis. It is probably safe to assume that the amount of lead found in your sample is below the maximum contaminant level. Breathe a sigh of relief. Color _____

IV. THE TEST FOR CALCIUM/MAGNESIUM

A. To test tube #9, add dropwise 0.1 M ammonium carbonate ($(NH_4)_2CO_3$). After each drop check for small white precipitate perhaps even crystalline slivers. This would be the formation of calcium carbonate ($CaCO_3$) or magnesium carbonate ($MgCO_3$). Sometimes these particles are very small and hard to detect. You might have to rotate the test tube in the light to catch a glimpse of the crystals. Do not add too much ammonium carbonate, this will re-dissolve the particles back into solution. Repeat this test with your water sample, test tube #4. What do you observe? _____

_____

_____

B. If the first attempt fails, inform your instructor. The instructor will demonstrate the appearance of the solids with a solution of calcium or magnesium nitrate and ammonium carbonate. Once you know what you are looking for try step A again on test tube #4.

C. Write a balanced chemical equation for the reaction of calcium nitrate with ammonium carbonate and for the reaction of magnesium nitrate with ammonium carbonate. Since two of the ions present in solution are called "spectator ions", re-write the two equations as net ion equations for Question 3 on the report page (page 4).

V. THE TEST FOR SODIUM

A. Obtain a tungsen or nichrome wire and bend a small loop at one end. First clean the wire by dipping it in HCl then burn off the acid using a bunsen burner. Compare the color of the flame as the acid is being burned off with the color of the flame after the wire has been heated to red-hot.

B. Dip the wire in a known solution of sodium chloride and perform the flame test on it. Report the color used to identify the presence of sodium. Clean the wire with HCl again. Dip the wire in test tube #5. Note the color of the flame. Is sodium present? **Yes** or **No**? (Circle one)

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1. Which ions did you find in your water sample? _____

2. Was the analysis of water in this experiment representative of a **physical** or **chemical** change? (Circle one)

3. Write balanced molecular equations describing your results from selective precipitation of the known samples. (see instructor for detail).

   I C. _____

   II B. _____

   III A. _____

   IV C. _____

   IV C. _____

4. Only for the ions found by selective precipitation, write the net ionic equations for all of the ions actually found in your water sample.