Experiment 5

Qualitative Analysis of Cations in Solution

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

- Observe chemical properties of barium, calcium, and magnesium ions in solution.
- Analyze an unknown solution and determine if it contains one or more of the following ions: barium, calcium, and magnesium.

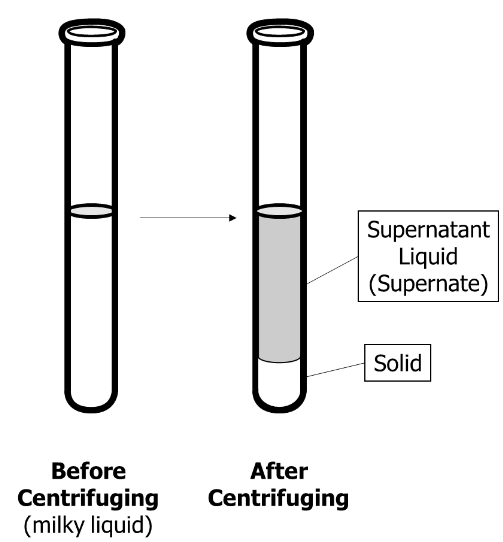
Background

Qualitative analysis is the systematic separation and identification of the chemical components in an unknown sample. In this experiment you will analyze an aqueous solution for Ba^{2+}, Ca^{2+}, and Mg^{2+}. An **aqueous solution** is homogenous mixture in which water has dissolved one or more substances.

Techniques used in this experiment

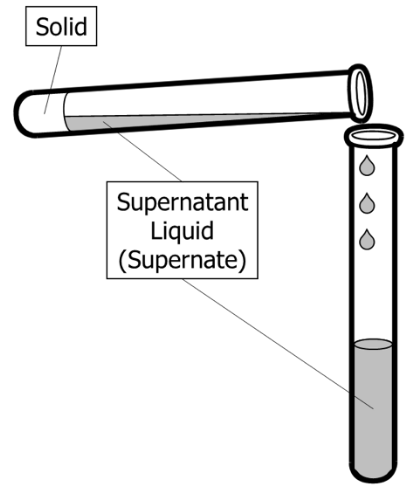
Precipitation of Ions
We can isolate one type of ion in a solution by adding a reactant that has been specially chosen to react with that ion to form a **precipitate** but will not react with the other ions if they are present. A **precipitate** is an insoluble solid that is formed when two aqueous solutions are mixed. For example, if ammonium sulfate is added to a solution containing Ba^{2+}, Ca^{2+}, and Mg^{2+}, the sulfate ions will only react with Ba^{2+} to form barium sulfate, but the sulfate ions will *not* react with Ca^{2+} or Mg^{2+}. The Ba^{2+} has been isolated (or removed) from the solution as solid barium sulfate. The solid can then be tested for barium to confirm the ion is present.

Centrifuging
When a precipitate is formed, it will cause the reaction mixture to look milky, cloudy, or hazy. If we wait long enough, all the solid will settle at the bottom of test tube. To speed up this process we will use a **centrifuge** to quickly separate the solid particles from the liquid. A centrifuge is an instrument that spins test tubes at a high speed to separate the solid from the liquid using centrifugal force. After centrifuging, the clear liquid that lies on top of the solid is called the supernatant liquid or **supernate**.



Decanting

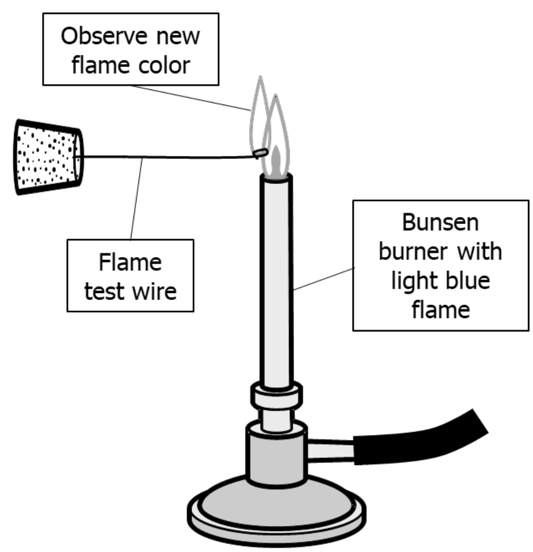
To decant a liquid is to carefully pour a liquid from one container to another without disturbing the solid at the bottom of the container. You may have used this technique in your daily life to separate coffee or tea from grounds or leaves at the bottom of a mug!



Decanting a Liquid

Flame tests

In this experiment we will perform a flame test to confirm the identity of the cation we have removed from the solution by turning it into a solid. The **flame test** is a method for identifying an ion using the unique color the ion emits when placed in a flame. To perform a flame test, you will dissolve the solid in dilute HCl, dip the looped end of a clean flame test wire into the solution so that a drop of liquid is caught in the loop, and place the loop in the edge of a Bunsen burner flame. You will then observe and record the flash of colored flame that is produced. For example, solution of barium ions will produce a flash of green flame. Flame tests should always be performed in a fume hood.

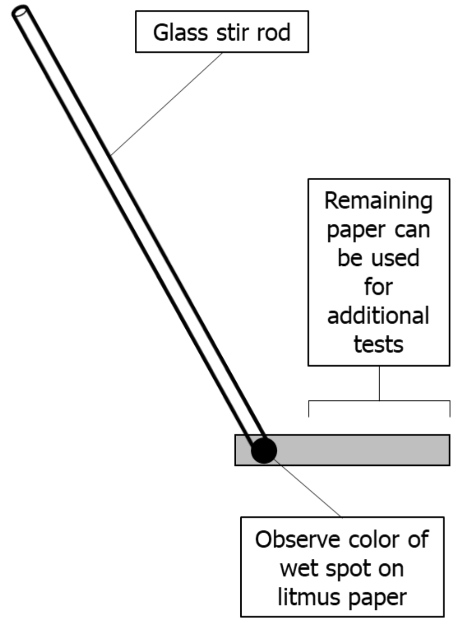


Performing a Flame Test

Testing for Acidity or Basicity

Litmus paper can be used to determine if a solution is acidic or basic. Dip the end of a clean glass stir rod into the solution and tap off any excess liquid. Touch the end of the glass stir rod to the litmus paper and observe the color of the wet spot.

Acidic solutions: red spot on blue litmus paper *Basic solutions:* blue spot on red litmus paper



Procedure

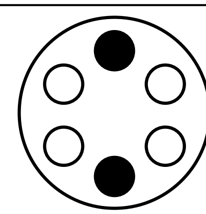
Safety and Waste Disposal

- Wear your goggles at all times.

- Avoid contact with all solutions. Many are irritants and some can cause chemical burns. If a solution gets on your skin, immediately rinse the affected area with water for several minutes and have your partner inform your instructor.

- All reactions must be disposed of in the hazardous waste jug for this experiment. Keep a temporary waste beaker at your station to collect completed reactions. At the end of the experiment, empty your temporary waste beaker into the hazardous waste jug in the fume hood.

- ALWAYS LOAD THE CENTRIFUGE EVENLY. Place test tubes containing mixtures across from each other to make sure the load is balanced. If you do not have more than one mixture to centrifuge, you can balance the load by using a second test tube filled to the same level with water. Unbalanced centrifuges will shake violently which can break test tubes, damage the centrifuge, injure you if the centrifuge breaks, and/or cause the centrifuge to fall off the counter!



Centrifuge loading pattern for two test tubes (black circles) or four test tubes (white circles).

- Lab centrifuges spin at a very high speed. Wait for the centrifuge to stop spinning on its own before removing a test tube your sample. Do NOT use your hand to stop the centrifuge. Newer centrifuges may stay locked shut until the rotor has stopped spinning.

Analysis of Known Sample

Important! Before using (or reusing) test tubes during this experiment, wash them with soap and water and then rinse with DI water to remove any traces of tap water. Tap water contains ions that will contaminate your sample and may give incorrect results.

1. Clean three small test tubes and a glass stir rod with soap and water. Rinse the test tubes and stir rod with DI water. Don't skip this step! Although the test tubes might look clean, they may still contain an invisible residue from a previous experiment that could give incorrect results.

2. Use a pencil to label the test tubes #1, #2, and #3 on the white marking spots. Take out a 250-mL beaker from your drawer and use a pencil to label it as your temporary waste container on the large white marking spot.

3. *Identifying Ba^{2+} in the known solution*
   a. Add 20 drops of the known solution to test tube #1. Add 20 drops of ammonium sulfate ($(NH_4)_2SO_4$) and mix with a glass stir rod.

      - If a white precipitate forms, Ba^{2+} might be present. In step 3c you will use a flame test to confirm that Ba^{2+} is present in the known mixture.

   b. Centrifuge the mixture for 60 seconds so that all of the white solid is at the bottom of the test tube. Carefully decant the supernate into the test tube #2 and save for step 4. The solid should stay in the bottom of test tube #1.

   c. Confirm the presence of Ba^{2+} by performing a flame test: Add 5 drops of dilute hydrochloric acid (HCl) to the solid in test tube #1 and stir the mixture well. Clean a flame-test wire by swishing it in a small test tube containing about 1 mL of dilute HCl. Tap off any excess liquid back into the test tube. Dip the looped end of the clean flame test wire into the mixture, making sure you catch a drop of liquid in the loop. Place the loop into the edge of a Bunsen burner flame and record the color of the flame. All flame tests should be performed in the fume hood.

      - A green flame confirms that Ba^{2+} is present in the known solution.

4. *Identifying Ca^{2+} in the known solution*
   a. Add 10 drops of ammonium oxalate ($(NH_4)_2C_2O_4$) to the solution in test tube #2 and mix.

      - If a white precipitate forms, Ca^{2+} might be present. In step 4d you will use a flame test to confirm that Ca^{2+} is present in the known mixture.

   b. Centrifuge the mixture for 60 seconds. Add one more drop of ammonium oxalate. If the liquid stays clear, go on to step 4c. If the liquid becomes cloudy again, add 2 more drops of ammonium oxalate and centrifuge for 60 seconds.

   c. Carefully decant the supernate into the test tube #3 and save for step 5. The solid should stay in the bottom of test tube #2.

   d. Confirm the presence of Ca^{2+} by performing a flame test: Add 5 drops of dilute hydrochloric acid (HCl) to the solid in test tube #2 and stir the mixture well. Clean a flame-test wire by swishing it in a small test tube containing about 1 mL of dilute HCl. Tap off any excess liquid back into the test tube. Dip the looped end of the clean flame test wire into the mixture, making sure you catch a drop of liquid in the loop. Place the loop into the edge of a Bunsen burner flame and record the color of the flame

- A brick-red flame confirms that Ca^{2+} is present in the known solution.

5. *Identifying Mg^{2+} in the known solution*
   a. Add 10 drops of sodium hydrogen phosphate (Na_2HPO_4) to the solution in test tube #3 and mix.

      - If a white precipitate forms, Mg^{2+} might be present. In step 5c you will use the blue lake test to confirm that Mg^{2+} is present in the known mixture.

   b. Centrifuge the mixture for 60 seconds. Carefully decant the supernate into your temporary waste beaker. The solid should stay in the bottom of test tube #3.

   c. Confirm the presence of Mg^{2+} by performing the blue lake test: Add 5 drops of magnesium indicator to the solid in test tube #3 and stir the mixture well. Add sodium hydroxide (NaOH) dropwise until the solution turns red litmus paper blue. Centrifuge the mixture and record your observations about the contents of test tube #3.

      - A blue gel precipitate at the bottom of the test tube confirms that Mg^{2+} is present in the known solution. If a blue gel does not form, add more NaOH, mix well, and centrifuge.

6. Empty the contents of test tubes #1, #2, and #3 in the waste beaker at your station. Clean all the test tubes with soap and water and rinse with DI water. You do NOT need to dry the test tubes before continuing to the next step.

Analysis of Unknown Sample

Analysis for Ba^{2+} in the unknown solution

7. Add 20 drops of the unknown solution to test tube #1. Add 20 drops of ammonium sulfate ($(NH_4)_2SO_4$) and mix with a glass stir rod.

- If there is no precipitate, Ba^{2+} is absent in the unknown. Transfer the liquid from test tube #1 to test tube #2 and *skip to step 8.*
- If a white precipitate forms, Ba^{2+} might be present. In step 7c you will use a flame test to confirm if Ba^{2+} is present in the unknown mixture.

b. Centrifuge the mixture for 60 seconds so that all of the white solid is at the bottom of the test tube. Carefully decant the supernate into the test tube #2 and save for step 8. The solid should stay in the bottom of test tube #1.

c. Confirm the presence of Ba^{2+} by performing a flame test: Add 5 drops of dilute hydrochloric acid (HCl) to the solid in test tube #1 and stir the mixture well. Clean a flame-test wire by swishing it in a small test tube containing about 1 mL of dilute HCl. Tap off any excess liquid back into the test tube. Dip the looped end of the clean flame test wire into the mixture, making sure you catch a drop of liquid in the loop. Place the loop into the edge of a Bunsen burner flame and record the color of the flame

- A green flame confirms that Ba^{2+} is present in the unknown solution.

8. *Analysis for Ca^{2+} in the unknown solution*

a. Add 10 drops of ammonium oxalate ($(NH_4)_2C_2O_4$) to the unknown solution in test tube #2 and mix.

- If there is no precipitate, Ca^{2+} is absent in the unknown. Transfer the liquid from test tube #2 to test tube #3 and *skip to step 9.*
- If a white precipitate forms, Ca^{2+} might be present. In step 8d you will use a flame test to confirm if Ca^{2+} is present in the unknown mixture.

b. Centrifuge the mixture for 60 seconds. Add one more drop of ammonium oxalate. If the liquid stays clear, go on to step 8c. If the liquid becomes cloudy again, add 2 more drops of ammonium oxalate and centrifuge for 60 seconds.

c. Carefully decant the supernate into the test tube #3 and save for step 9. The solid should stay in the bottom of test tube #2.

d. Confirm the presence of Ca^{2+} by performing a flame test: Add 5 drops of dilute hydrochloric acid (HCl) to the solid in test tube #2 and stir the mixture well. Clean a flame-test wire by swishing it in a small test tube containing about 1 mL of dilute HCl. Tap off any excess liquid back into the test tube. Dip the looped end of the clean flame test wire into the mixture, making sure you catch a drop of liquid in the loop. Place the loop into the edge of a Bunsen burner flame and record the color of the flame

- A brick-red flame confirms that Ca^{2+} is present in the unknown solution.

9. *Analysis for Mg^{2+} in the unknown solution*
   a. Add 10 drops of sodium hydrogen phosphate (Na_2HPO_4) to the unknown solution in test tube #3 (or test tube #1 or #2 depending on whether Ba^{2+} and/or Ca^{2+} are absent) and mix.

      - If there is no precipitate, Mg^{2+} is absent in the unknown. *You are done analyzing your unknown solution.*
      - If a white precipitate forms, Mg^{2+} might be present. In step 9c you will use the blue lake test to confirm if Mg^{2+} is present in the unknown mixture.

   b. Centrifuge the mixture for 60 seconds. Carefully decant the supernate into your waste beaker. The solid should stay in the bottom of test tube #3.

   c. Confirm the presence of Mg^{2+} by performing the blue lake test: Add 5 drops of magnesium indicator to the solid in test tube #3 and stir the mixture well. Add sodium hydroxide (NaOH) dropwise until the solution turns red litmus paper blue. Centrifuge the mixture and record your observations about the contents of test tube #3.

      - A blue gel precipitate at the bottom of the test tube confirms that Mg^{2+} is present in the unknown. If a blue gel does not form, add more NaOH, mix well, and centrifuge.

10. Empty all test tubes into the temporary waste beaker at your station. Dispose of the contents of your temporary waste beaker in the hazardous waste jug in the fumehood. Wash all glassware and return it to your drawer.

Experiment 5- Report Sheet

Name ____ **Date** _______________________

Partner's Name ________________________________ **Lab: M T W R at** _____________

Unknown Solution ID ___________

Record your observations for each of the steps in the table below.

Test tube #1: Separation and testing for Ba^{2+}

	Known Solution	**Unknown Solution**
After adding ammonium sulfate		
Flame test Color		

Test tube #2: Separation and testing for Ca^{2+}

	Known Solution	**Unknown Solution**
After adding ammonium oxalate		
Flame test Color		

Test tube #3: Separation and testing for Mg^{2+}

	Known Solution	**Unknown Solution**
After adding ammonium hydrogen phosphate		
Blue lake test results		

Unknown Solution ID ___________

The cation(s) present in my solution: _________________

The cation(s) absent in my solution: ___________

Experiment 5—Post-Lab Assignment

Name: ____

1. Which cations (Ba^{2+}, Ca^{2+}, Mg^{2+}) are present and absent in an unknown solution given the following observations?
   - The unknown solution in test tube #1 plus $(NH_4)_2SO_4$ gives a white precipitate.
   - The supernate in test tube #1 is poured into test tube #2.
   - The flame test of the white precipitate remaining in test tube #1 produces a green flame.
   - The solution in test tube #2 plus $(NH_4)_2C_2O_4$ gives a white precipitate.
   - The supernate in test tube #2 is poured into test tube #3.
   - The flame test of the white precipitate remaining in test tube #2 produces a brick red flame.
   - The solution in test tube #3 plus Na_2HPO_4 and NaOH gives no reaction.

   Cation(s) present ________________ Cation(s) absent ________________

2. Which cations (Ba^{2+}, Ca^{2+}, Mg^{2+}) are present and absent in an unknown solution given the following observations?
   - The unknown solution in test tube #1 plus $(NH_4)_2SO_4$ gives no reaction.
   - The solution in test tube #1 is poured into test tube #2.
   - The solution in test tube #2 plus $(NH_4)_2C_2O_4$ gives a white precipitate.
   - The supernate in test tube #2 is poured into test tube #3.
   - The flame test of the white precipitate remaining in test tube #2 produces a brick red flame.
   - The solution in test tube #3 plus Na_2HPO_4 and NaOH gives a white precipitate.
   - The white precipitate in test tube #3 dissolves in HCl; magnesium indicator and NaOH is added until the solution tests basic. A blue gel is observed at the bottom of the test tube after centrifuging.

   Cation(s) present ________________ Cation(s) absent ________________

3. Write the names for the following ions using the Stock system. The first one has been done for you as an example.

   a. Cu^{+} copper(I) ion ____________ b. Cu^{2+} ______________________________

   c. Fe^{2+} ______________________________ d. Fe^{3+} ______________________________

   e. Sn^{2+} ______________________________ f. Sn^{4+} ______________________________

4. Complete the table below as shown by the example. Give the correct formula and name or the compounds using the ions.

	NO_3^-	SO_4^{2-}	PO_4^{3-}
Ba^{2+}	$Ba(NO_3)_2$ barium nitrate		
Ca^{2+}			
Mg^{2+}			

5. Complete the table below as shown by the example. Give the correct formula and name or the compounds using the ions.

	nitrite ion	sulfite ion	phosphite ion
mercury(II) ion	$Hg(NO_2)_2$ mercury(II) nitrite		
iron(III) ion			
lead(IV) ion			

Name: ______________________________________

Pre-Lab Assignment for Qualitative Analysis of Cations in Solution

1. Match the following terms to their definitions: Aqueous solution, cation, centrifuge, decant, flame test, precipitate, qualitative analysis, supernate

Definition	Term
a. an ion with a positive charge	
b. the systematic separation and identification of the chemical components in an unknown sample	
c. a solution of one (or more) substances dissolved in water	
d. an insoluble solid formed when two solutions are mixed	
e. an instrument that spins test tubes to separate solids from liquids	
f. the liquid above a solid after a mixture has been centrifuged	
g. the process of pouring a liquid from one container to another without transferring the solid at the bottom of the container	
h. a method for identifying an ion using the color the ion emits when placed in a flame	

2. What are the three cations you will study in this experiment?

3. If you put a drop of basic solution on red litmus paper, what will be the color of the wet spot?

4. Where should you place the looped end of the wire during a flame test?

5. Read the procedure carefully to answer the following questions.

   a. Which cation is confirmed in test tube #1 by a green flame test? ____________________

   b. Which cation is confirmed in test tube #2 by a brick-red flame test? _________________

   c. Which cation is confirmed in test tube #3 by a blue gel precipitate? __________________

6. Why is it necessary to wash all test tube with soap and water and rinse with deionized (DI) water before using them in this experiment?

7. Use the following observations and refer to the steps 6 through 9 of the procedure to determine which cations (Ba^{2+}, Ca^{2+}, Mg^{2+}) are present and absent in an unknown solution.
   a. The unknown solution in test tube #1 plus $(NH_4)_2SO_4$ gives a white precipitate.
   b. The supernate in test tube #1 is poured into test tube #2.
   c. The flame test of the white precipitate remaining in test tube #1 produces a green flame.
   d. The solution in test tube #2 plus $(NH_4)_2C_2O_4$ gives a white precipitate.
   e. The supernate in test tube #2 is poured into test tube #3.
   f. The flame test of the white precipitate remaining in test tube #2 produces a brick red flame.
   g. The solution in test tube #3 plus Na_2HPO_4 and NaOH gives a white precipitate.
   h. The white precipitate in test tube #3 dissolves in HCl; magnesium indicator and NaOH is added until the solution tests basic. A blue gel is observed at the bottom of the test tube after centrifuging.

   Cation(s) present _________________ Cation(s) absent _________________

8. Use the following observations and refer to the steps 6 through 9 of the procedure to determine which cations (Ba^{2+}, Ca^{2+}, Mg^{2+}) are present and absent in an unknown solution.
   - The unknown solution in test tube #1 plus $(NH_4)_2SO_4$ gives no reaction.
   - The solution in test tube #1 is poured into test tube #2.
   - The solution in test tube #2 plus $(NH_4)_2C_2O_4$ gives no reaction.
   - The solution in test tube #2 is poured into test tube #3.
   - The solution in test tube #3 plus Na_2HPO_4 and NaOH gives a white precipitate.
   - The white precipitate in test tube #3 dissolves in HCl; magnesium indicator and NaOH is added until the solution tests basic. A blue gel is observed at the bottom of the test tube after centrifuging.

   Cation(s) present _________________ Cation(s) absent _________________

9. What safety precautions should be taken while performing this experiment?