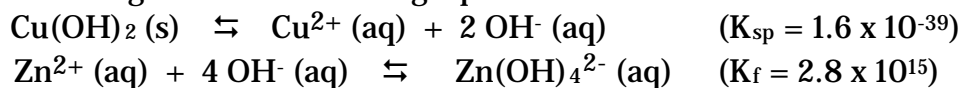


CATION GROUP SEPARATION

INTRODUCTION

Qualitative analysis is a procedure for identifying substances in a mixture. In this experiment you will be identifying cations present in a solution. These ions are identified by specific chemical tests but because one cation can interfere with a test for another ion, the ions must first be separated. By taking advantage of the differences in solubility, groups of ions are selectively separated from other ions in the solution through the use of group reagents which form precipitates with given groups. For example, when dilute hydrochloric acid is added to a mixture of cations, only the chlorides of lead (II), silver, and mercury (I) precipitate because all other chlorides are soluble. These three metal ions are classified as members of Group I and hydrochloric acid is the group reagent. After separation from the Group I precipitate, the solution (supernatant) is then treated with the Group III reagent and conditions and the members of that group will precipitate out. NaOH is the group reagent used in Groups II and III and separation occurs by controlling the pH. Separation is carried out making use of the following equilibrium:



Under the conditions used, the concentration of hydroxide ion (from aqueous NH_3) is sufficient to precipitate out Cu^{2+} as a hydroxide (which are very insoluble) and the concentration of OH^- is in excess and is large enough to produce $\text{Zn(OH)}_3^{2-} (\text{aq})$ as a complex ion

In today's experiment, a mixture containing a representative cation from each group is analyzed.

The ions found in the various cation groups are:

**Note: The ions indicated with a Roman numeral probably do not exist as simple ions in solution and the Roman numeral given is the oxidation state.*

Group I	$\text{Ag}^+, \text{Pb}^{2+}, \text{Hg}_2^{2+}$
Group II	$\text{Pb}^{2+}, \text{Bi}^{3+}, \text{Cu}^{2+}, \text{As}^{\text{III}}, \text{As}^{\text{V}}, \text{Sb}^{\text{III}}, \text{Sb}^{\text{V}}, \text{Sn}^{\text{II}}, \text{Sn}^{\text{IV}}$
Group III	$\text{Fe}^{2+}, \text{Fe}^{3+}, \text{Al}^{3+}, \text{Co}^{2+}, \text{Ni}^{2+}, \text{Mn}^{2+}, \text{Zn}^{2+}, \text{Cr}^{3+}$
Group IV	$\text{Ba}^{2+}, \text{Sr}^{2+}, \text{Ca}^{2+}$
Group V	$\text{Na}^+, \text{K}^+, \text{NH}_4^+, \text{Mg}^{2+}$

In this experiment you will be dealing with only one representative of each cation group.

*** SAFETY:**

- Safety goggles must be worn at all times
- AgNO_3 will stain your hands black. Rinse your hands with tap water after handling.
- Dilute hydrochloric acid (HCl) and aqueous ammonia ($\text{NH}_3 (\text{aq})$) can harm eyes, skin, and clothing. Handle with care. Any acid spilled on the skin or splashed into your eyes must be rinsed with a large volume of water. Wash your eyes at the eye wash station.

*** DISPOSAL:**

- Dispose the Ag reaction mixture in the specially labeled for cation group separation "Silver, Ag" container and not in the sink.
- Dispose the Cu(OH)_2 and Zn(OH)_2 reaction mixtures in the specially labeled waste container cation group separation Cu(OH)_2 and Zn(OH)_2 and not in the sink.
- Dispose the Ba reaction mixture in the specially labeled waste container cation group separation, Ba mixture and not in the sink!

LAB NOTEBOOK SET-UP

- **Purpose**
- **Materials and Equipment Table**
- **Flow Chart**
- **Safety**
- **Disposal**
- **Procedure**
- **Questions**
- **Summary**

PROCEDURE

A. Sample Preparation

Mix together in a single 4 inch test tube the following 0.1 M solutions of the following cations: 4 drops of Ag^+ , Cu^{2+} , Na^+ , and Zn^{2+} , 12 drops of Ba^{2+} and K^+ (As AgNO_3 , $\text{Cu}(\text{NO}_3)_2$, NaNO_3 , $\text{Zn}(\text{NO}_3)_2$, and $\text{Ba}(\text{NO}_3)_2$, KNO_3 respectively.)

B. Group I Separation

Add 4 drops of 6 M HCl. Mix well, heat in the hot water bath to just below boiling for 10 minutes. Centrifuge. (Make sure you use a counter-balance when you centrifuge). A precipitate indicates, Ag^+ presence, a Group I ion. Test for complete precipitation by adding a drop of 6 M HCl to the supernatant. Make and record observations for both the supernatant and precipitate. Write the net-ionic equation for the reaction. (This is a simplified procedure adapted to the 5 ions present. If you were running a general unknown with a multiple number of unknown cations included, the procedure involving sub-groups and final confirmation tests for each of the ions would be followed under each group.)

* **DISPOSAL:** Dispose the Ag reaction mixture in the specially labeled for cation group separation "Silver, Ag" container and not in the sink!

C. Group II Separation

Transfer the supernatant to another test tube. Add 6 M NaOH until basic (pH= 8-10) to universal pH paper. Mix well. Add 4 drops of 6 M NaOH in excess. Mix well, heat in the hot water bath to just below boiling for 5 minutes. Centrifuge. (Make sure you use a counter-balance when you centrifuge). If the precipitate is deep blue, Cu^{2+} , a cation of Group II, is present as $\text{Cu}(\text{OH})_2$, or if you obtained black ppt, CuO . Make and record observations for both the supernatant and precipitate. Write the net-ionic equation for the reactions.

* **DISPOSAL:** Dispose the $\text{Cu}(\text{OH})_2$ reaction mixture in the specially labeled waste container cation group separation $\text{Cu}(\text{OH})_2$ and $\text{Zn}(\text{OH})_2$ and not in the sink!

D. Group III Separation

With a disposable pipet, transfer the supernatant to another test tube. Add 6M aq HCl until the solution is acid to universal indicator paper (i.e. pH of 2-4). Then add two "pea size" amount of $\text{NaC}_2\text{H}_3\text{O}_2$. Add 6 M NaOH until the solution is basic (pH= 8-10) to universal pH paper. (Warning! Do not make the pH any higher than a pH =11. Mix well, heat in the hot water bath for 5 minutes. Centrifuge. A precipitate indicates, Zn^{2+} presence as $\text{Zn}(\text{OH})_2$, a Group III ion. Make and record observations. Write the net-ionic equation for the reactions

* **DISPOSAL:** Dispose the $\text{Zn}(\text{OH})_2$ reaction mixture in the specially labeled waste container cation group separation $\text{Cu}(\text{OH})_2$ and $\text{Zn}(\text{OH})_2$ and not in the sink!

E. Group IV Separation

Transfer the supernatant to another test tube. Add 6 M H_2SO_4 dropwise until the solution is acidic (pH is 2-4). Heat to just below boiling in the water bath. A precipitate indicates

Ba²⁺ presence, a cation of Group IV. Centrifuge. Make and record observations. Write the net-ionic equation for the reactions.

* **DISPOSAL:** Dispose the Ba reaction mixture in the specially labeled waste container cation group separation, Ba mixture and not in the sink!

F. Group V Potassium Flame Test

Transfer the supernatant to a crucible and concentrate the solution to a volume of approximately .5 ml (approximately 3 mm in your crucible) by heating over a Bunsen burner. Perform a flame test for potassium using the cobalt glass in your locker.

Flame Test:

1. Make a small loop at the end of the Pt wire
2. Clean the Pt wire by dipping the loop into conc HCl and then into the hottest part of the Bunsen burner flame. (Repeat this step several times)
3. Dip the wire into your sample and place the Pt wire into the hottest part of the flame.
4. To see the K⁺ flame, you will need to look at the flame thru a cobalt glass.

Make and record observations (Include the color of the flame as well as the length of time of the flame) Write equations for both the Na and K flame tests.

Questions

1. Explain fully using equilibrium, why the pH needs to be above 8 and an excess of NaOH added for to separate cation II, Cu (II) and cation III, Zn²⁺.
2. Explain why pH in Group III separation, the pH must be between 8-10 and no higher.

Summary

Write a summary summarizing this experiment. Include key points as well as supporting statements.