Experiment 9.

Qual I. Na^+, K^+, NH_4^+, Mg^{2+}, Ca^{2+}, Cu^{2+}

Experimental Procedure

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OBJECTIVES

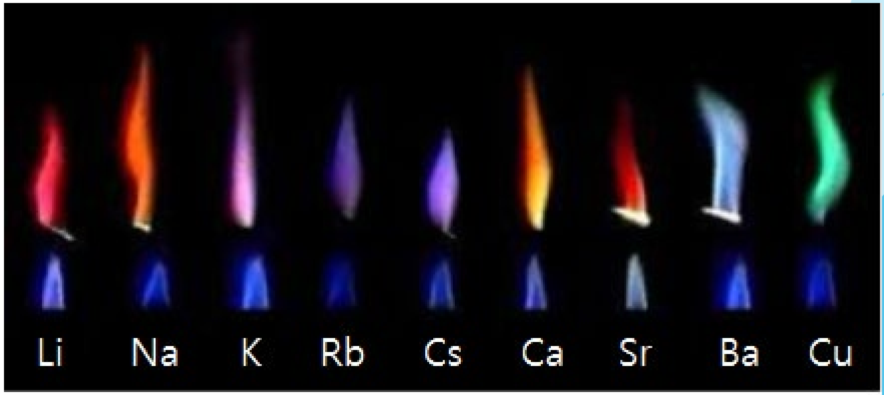
- To observe and utilize the chemical and physical properties of Na^+, K^+, NH_4^+, Mg^{2+}, Ca^{2+}, and Cu^{2+}
- To separate and identify the presence of one or more of the cations, Na^+, K^+, NH_4^+, Mg^{2+}, Ca^{2+}, and Cu^{2+}

INTRODUCTION

Flame Test

Flame test is a simple and effective chemical experiment used to identify the presence of metal ions. When a sample containing metal ions is introduced to a flame, each type of metal ion emits a specific color.

This occurs because the heat excites the electrons of the metal ions, causing them to jump to higher energy levels and then fall back down, releasing light at characteristic wavelengths. The flame test is primarily used for qualitative analysis, providing a quick way to confirm the presence of specific metal ions.



Ionic Equilibria

Ionic equilibria refer to the state of balance that exists in a solution where ionic compounds dissociate into their respective ions. When an ionic compound dissolves in water, it separates into positive and negative ions. The equilibrium is established when the rate of dissociation of the compound into ions equals the rate of recombination of the ions back into the compound. This concept is fundamental in understanding acid-base reactions, solubility, and buffer solutions.

Acid–Base Control

Acid–base control refers to the regulation of pH in a solution through the use of acids, bases, and buffer solutions. Acids release hydrogen ions (H^+) in solution, while bases accept hydrogen ions or release hydroxide ions (OH^-). The pH scale measures the concentration of hydrogen ions, determining the acidity or basicity of a solution.

Complex Formation

Complex formation involves the creation of a coordination complex, where a central metal ion binds to one or more molecules or ions, called ligands, through coordinate covalent bonds. These complexes can have various shapes and properties depending on the metal ion and the ligands involved.

EXPERIMENTAL PROCEDURE

Procedure Overview

Two solutions are tested wit various reagents in this analysis:

(1) A reference solution containing all six of the cations of I and (2) a test solution containing any number of Qual I cations. Separations and observations are made and recorded. Equations that describe the observations are also recorded. Comparative observations of the two solutions results on the identification of the cations in the test solution. All tests are qualitative; only identification of the cations(s) is required. To simply the analysis, take the following steps:

1. Reference solution: At each circled superscript, stop and record data on the ***Report Sheet***. After the presence of a cation is confirmed, save the characteristic appearance of the cation in the test tube so that it can be compared with observations made in the analysis of your test solution.

2. Test solution: Simultaneously perform the same procedure on the test solution and make a comparative observation. Check the findings on the ***Report Sheet***. Do not discard any solutions (but keep all solutions labeled) until the experiment is complete. Record the test solution number on the Report Sheet.

A. Test for Sodium Ion

1. Remove the interfering ions. Place no more than 2 mL of the reference solution an evaporating dish. Add a "pinch" or two of solid $(NH_4)_2C_2O_4$ (Caution: *avoid skin contact*), with stirring, until the solution is basic to pH paper; add a slight excess of the solid and then a pinch of solid $(NH_4)_2CO_3$. Heat the solution slowly in a fume hood (NH_3 fumes may be evolved) to a moist residue, not to dryness! Allow the evaporating dish to cool. Add up to 1 mL of distilled water, stir, and decant into your smallest beaker.

2. Confirmatory test. The flame test for sodium ions is reliable but also requires some technique. Clean the flame test wire by dipping it in 6 M HCl (*Caution!*) and heating it in he hottest part of a Bunsen flame until the flame is colorless.

Repeat as necessary. Dip the flame test wire into the solution in the beaker and place it in the flame. A brilliant yellow persistent flame indicates the presence of sodium. Conduct the sodium flame test on a 0.5M NaCl solution for comparison..

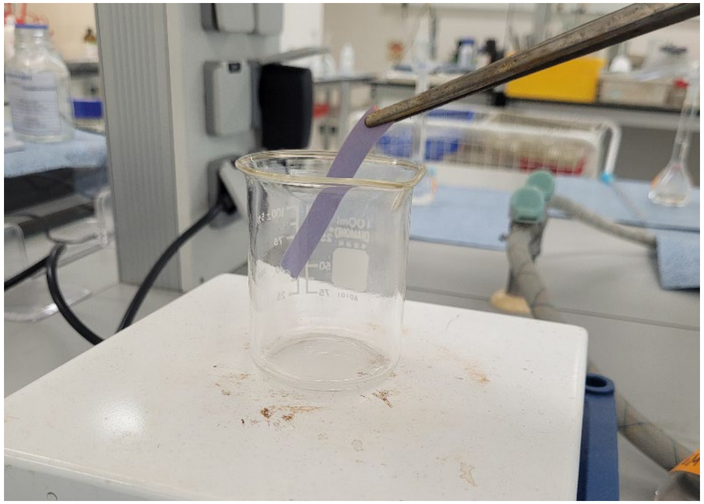
B. Test for Potassium Ion

Confirmatory Test. Repeat Part A.2. A fleeting lavender flame confirms the presence of potassium. If sodium is present, view the flame through cobalt blue glass/ Several trials are necessary as the test is judgmental. Conduct flame test on a 0.5 M KCl solution for comparison.

C. Test for Ammonium Ion

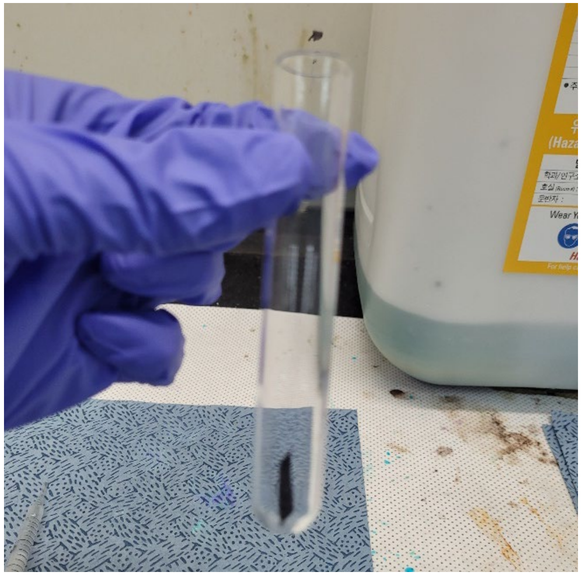
1. Prepare the sample. Transfer 5 mL of the original reference solution to a 100-mL beaker, support it on a wire gauze, and heat until a moist residue forms (do not evaporate to dryness!). Moisten the redisue with 1-2 mL of deionized water.

2. Confirmatory test. Moisten a piece of red litmus paper with water. Add 1-2mL of 6M NaOH to the reference solution, suspend the litmus above the solution, and very gently warm the mixture-do not boil. (*Caution: Be careful not to let the NaOH contact the litmus paper.*) A change in litmus from *red to blue* confirms the presence of ammonia. The nose is also a good detector, but it is not always as sensitive as the litmus test.

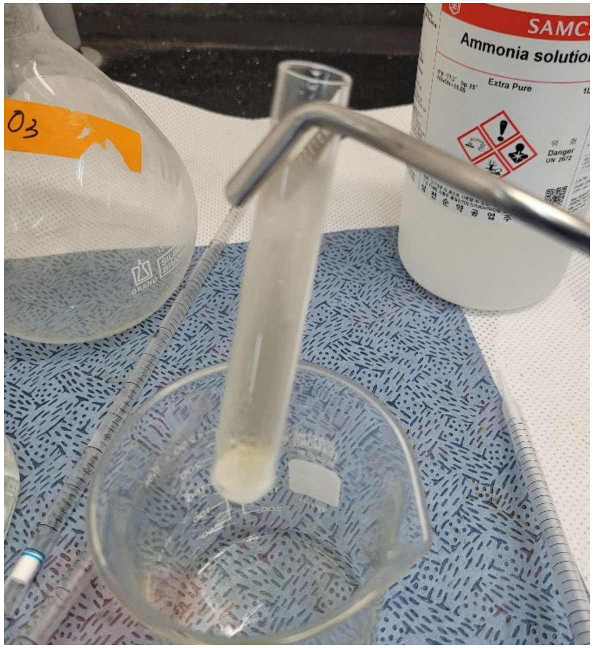


D. Test for Copper Ion

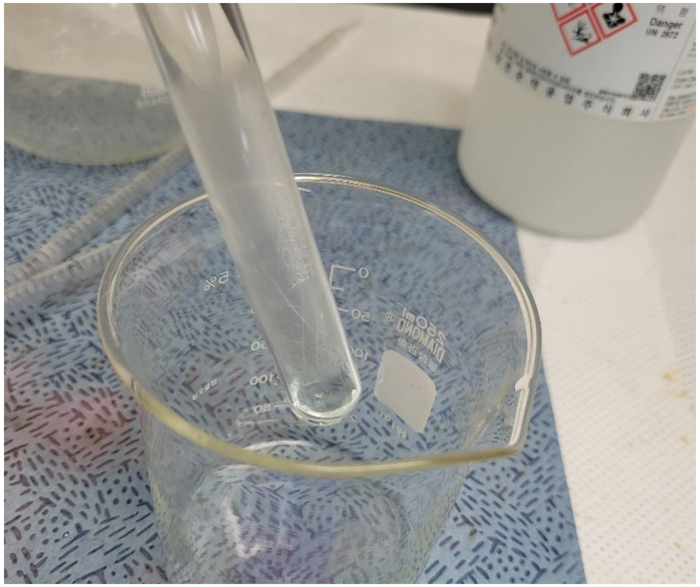
1. Reduction of copper(II) ion. Begin with 5 mL of the original reference solution in a 150-mm test tube. Polish a 1-cm strip and place it into the solution and let stand for 10-20 minute. Decant the solution and save for Part E.1. Wash the solid (now copper metal and excess zinc metal) with at least three portions of deionized water and discard the washings.



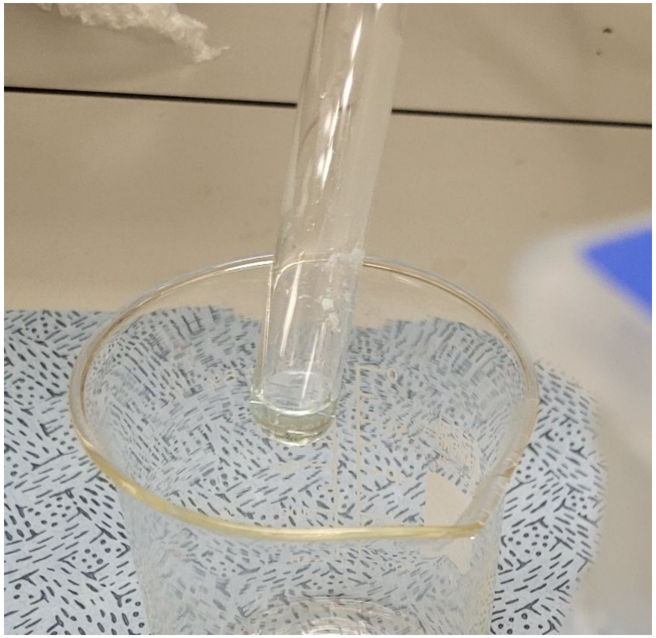
2. Oxidation of copper metal. Transfer the test tube containing the copper metal to a fume hood. Add drops of 6 M HNO_3 (**Caution**: 6 M HNO_3, *cause sever skin burns, and the evolved NO gas is toxic*) until the copper metal completely reacts. Be patient; some heating in a water bath may be necessary.

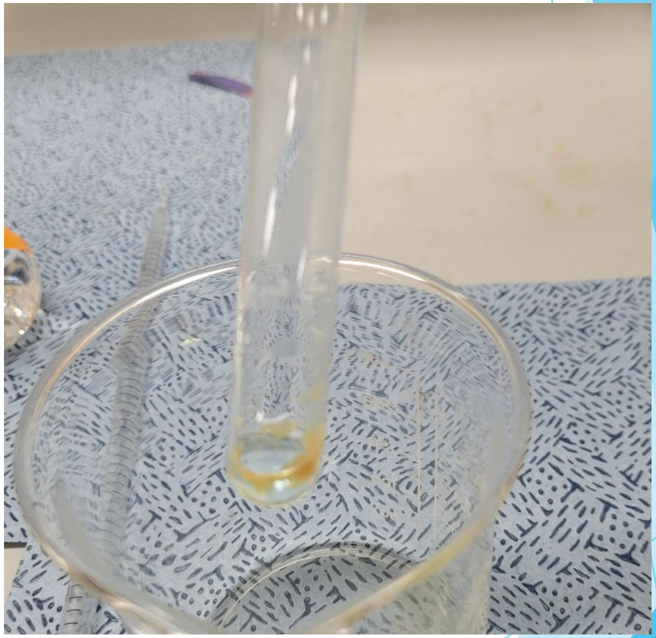


3. Confirmatory test. Slowly add drops of conc NH_3 (**Caution**: *Avoid inhalation or skin contact!*) to the solution from Part D.2. The deep-blue confirms the presence of Cu^{2+} in the solution.



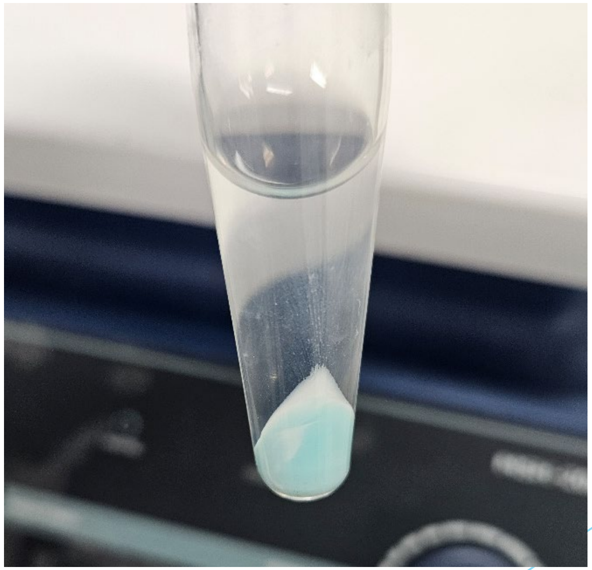
4. A second confirmatory test. Acidify the solution from Part D.3. to pH paper with 6 M CH_3COOH (**Caution!**). Add 3 drops of 0.2M $K_4[Fe(CN)_6]$. A red-brown precipitate reconfirms the presence of Cu^{2+} ion.





E. Test for Calcium Ion

1. Sample preparation. The supernatant from Part D.D contains Ca^{2+}, Mg^{2+}, and Zn^{2+} (From the reduction of Cu^{2+}). Add drops of 6 M NH_3 until the solution is just basic to pH paper. Add 2-3 drops of 1 M $K_2C_2O_4$. A white precipitate confirms the presence of Ca^{2+} and/or Zn^{2+} as both CaC_2O_4 and ZnC_2O_4 have marginal solubility. If no precipitate forms immediately, warm the solution in a water bath, cool, and let stand. Centrifuge and save the supernatant for Part F.



2. Confirmatory test. Wet the precipitate to a moist paste with a drop of 6 M HCl and perform a flame test. A fleeting yellow-red flame is characteristic of calcium ion and confirms its presence.

F. Test for Magnesium Ion

1. Confirmatory test. Add 1-2 drops of 6 M NH_3 to the supernatant from Part E.1. Add 2-3 drops of 1 M Na_2HPO_4, heat in a hot water (~90°C) bath, and allow to stand. The precipitate may be slow in forming; be patient. Observing the white precipitate confirms the presence of Mg^{2+} ion.

Cleanup

Rinse each test tube twice with tap water. Discard each rinse in the Waste Metal Salts container. Thoroughly clean each test tube with soap and tap water; rinse twice with tap water and twice with deionized water.

Disposal: Dispose of all test solutions and precipitate in the Waste Metal Salts container.