

Qualitative Ion Analysis

1.0 Introduction

Analytical chemistry is the branch of chemical studies that seeks to analyze unknown chemical substances. An **analytical chemist** who desires to analyze an known chemical will often be asking 1) what chemicals are present? and 2) what are their amounts? The first question is a qualitative question that is answered by **qualitative analysis**. The second question is quantitative and is answered by **quantitative analysis**. In our experiment we will be concerned with what ions are present in an unknown so we will use qualitative analysis to confirm the presence of various ions; however we will not know the quantities present.

The most common approach to analyzing a solution for the presence of various ions is to subject the solution to various **confirmatory tests**. These are chemical reactions which cause a distinct chemical change with a specific ion. Tests will often form precipitates, produce gases or cause color changes in the presence of a specific ion. To analyze a solution, the solution will be divided into small portions and various chemicals or **reagents** will be added. It is important to use fresh samples for each test so there will not be a buildup of too many ions in the test solution. If too many ions are present, it is possible to get various interference reactions. In this experiment the solutions will be prepared so that interferences will be minimized but in real world samples more advanced techniques are employed to overcome this difficulty.

2.0 PROCEDURE

A. First, using known samples you will practice the confirmatory test for specific ions. As you run each test make a note of the specific chemical change that occurs for each ion. Record your observations on the report sheet for reference. The test ion compound will be in **BOLD** letters and the chemical used for the test will be in *ITALICS*.

B. After all of the confirmatory tests are completed, you will analyze an unknown which will contain 2 to 3 of the ions. The unknown will be divided into fresh, small samples for each new test. The small portion of unknown will **REPLACE** the **BOLD** chemical in each of the confirmatory tests since we do NOT want to add the ion we are testing for!

CONFIRMATORY TESTS - DISCARD IN THE WASTE CONTAINER WHEN DONE

1. Carbonate ion CO_3^{2-}

Place **10 drops of 0.1 M Na_2CO_3** (sodium carbonate) in a test tube. **Carefully** add *10 drops of 6M HCl* (dilute hydrochloric acid) watching for the release of gas with the addition of each drop. A bubbling of carbon dioxide gas such as seen in carbonated beverages confirms the presence of carbonate ion.

2. Thiocyanate ion SCN^-

Place **10 drops of 0.1 M KSCN** (potassium thiocyanate) in a test tube. Add *10 drops of 6M $\text{HC}_2\text{H}_3\text{O}_2$* (dilute acetic acid) and stir the mixture. If upon adding *2 to 4 drops of 0.1 M $\text{Fe}(\text{NO}_3)_3$* a **deep red** color forms the presence of thiocyanate ion is confirmed.

Qualitative Ion Analysis

Experiment 6

3. Chloride ion Cl^-

Place **10 drops of 0.1 M NaCl** (sodium chloride) in a test tube. Next add 10 drops of 6 M HNO_3 and stir well. Carefully add *3 drops of 0.1 M AgNO_3* (silver nitrate). The formation of a white precipitate of AgCl confirms the presence of chloride ion.

4. Nickel ion Ni^{2+}

Place **10 drops of 0.1 M NiSO_4** (nickel sulfate) in a test tube. Add *2 drops of 1M NH_3* (ammonia or NH_4OH) followed *10 drops of 1% alcoholic dimethylglyoxime* and stir with a glass rod. A red, gelatinous precipitate will form if nickel ion is present.

5. Copper(II) ion Cu^{2+}

Place **10 drops of 0.1 CuSO_4** (copper(II) sulfate) in a test tube. In a fume hood, add *concentrated NH_3* (ammonia or 15M NH_4OH) drop by drop. The initial formation of a light blue precipitate which re-dissolves to form a dark blue solution confirms copper(II) ion.

6. Iron(III) ion Fe^{3+}

Place **10 drops of 0.1M $\text{Fe}(\text{NO}_3)_3$** (iron(III) nitrate) in a test tube. Add *4 drops of 0.1M KSCN* (potassium thiocyanate). A deep red color confirms iron(III) ion.

7. Unknown analysis

Get a small sample (5mL) of one of the unknowns and record its ID on your report sheet. The unknown will contain 2 or 3 of the ions tested above. Use **10 drop portions** of the unknown in place of the **BOLD** chemicals in each of the six tests above. For each test, mark on the report sheet whether the ion being tested for is present or absent. Again **discard each solution in the waste container when finished**. If you have trouble, with a test, you may want to repeat the known test for comparison. You can also make up a known solution which contains all of the ions you think are present and test it to see if it behaves like your unknown.

Qualitative Ion Analysis**Experiment 6****Report Sheet**

NAME _____

UNKNOWN ID _____

Ion Tested	Observations	Present in Unk.	Absent in Unk.
CO_3^{2-}			
SCN^-			
Cl^-			
Ni^{2+}			
Cu^{2+}			
Fe^{3+}			

Ions present in unknown :

QUESTION:

*** This is a hypothetical question and does NOT directly relate to any of your specific results.***

1) An unknown that might contain any of the six ions studied in this experiment (but no others) has the following test results:

- On addition of 6M HCl, no visible change occurs.
- When 0.5 M KSCN is added to the unknown, a deep red color appears.

On the basis of the preceding information, classify each of the following ions as present (P), absent (A) or undetermined (U) by the tests described:

 CO_3^{2-} _____ SCN^- _____ Cl^- _____

Ni^{2+} _____ Cu^{2+} _____ Fe^{3+} _____

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