Qualitative Analysis of Some Common Ions

Performance Goals

- 13–1 Conduct tests to confirm the presence of known ions in a solution.
- **13–2** Analyze an unknown solution for certain ions.

CHEMICAL OVERVIEW

When we analyze an unknown solution, two questions come to mind: (1) what ions are present in the solution and (2) what is their concentration? The first question can be answered by performing a **qualitative analysis**, and the second by a **quantitative analysis**. These two broad categories are known collectively as **analytical chemistry**. In this experiment, you will perform a qualitative analysis.

The general approach to finding out what ions are in a solution is to test for the presence of each possible component by adding a reagent that will cause that component, if present, to react in a certain way. This method involves a series of tests, one for each component, carried out on separate samples of solution. Difficulty sometimes arises, particularly in complex mixtures, because one of the species may interfere with the analytical test for another. Although interferences are common, many ions in mixtures can usually be identified by simple tests.

In this experiment, you will analyze an unknown mixture that may contain one or more of the following ions in solution:

$$CO_{3}^{2-}Cl^{-}SCN^{-}SO_{4}^{2-}Cl^{-}Cu^{2+}Al^{3+}$$

First, you will perform the various tests designed to detect the presence of individual ions. Once you have observed these specific reactions, you will obtain the unknown solution from your instructor. Then, taking small portions of this solution, you will run each reaction again to determine which ions are present and which are absent.

This experiment is designed to test the behavior of only a few ions. More complex schemes are used for a more complete qualitative analysis. **172** *Introduction to Chemical Principles: A Laboratory Approach* ■ Weiner and Harrison

SAFETY PRECAUTIONS AND DISPOSAL METHODS

In some tests you will be required to use fairly concentrated acids and bases. When in contact with skin, most of these chemicals cause severe burns if not removed promptly. Wear goggles when working with any of the reagents required in this experiment. Discard all solutions in the container provided. PROCEDURE A boiling water bath is required for some of the tests you are to perform. Pour about 100 mL of deionized water into a 150-mL beaker and heat it to boiling. Maintain it at that temperature throughout the experiment, replenishing the water from time to time as it becomes necessary. 1. Test for the Carbonate Cautiously add about 10 drops of 1 M hydrochloric acid, HCl, to 10 drops lon, CO₃² of 1 M sodium carbonate, Na₂CO₃, in a small size test tube. Bubbles of colorless and odorless gas of carbon dioxide, CO₂, usually appear immediately, indicating the presence of the carbonate ion. If the bubbles are not readily apparent, warm the solution in the hot water bath and stir. 2. Test for the Sulfate Ion, Cautiously add about 10 drops of 1 M hydrochloric acid, HCl, to 10 drops SO₄2 of 0.5 M sodium sulfate, Na₂SO₄. Then add 3 to 4 drops of 1 M barium chloride, BaCl₂. A white, powdery precipitate of barium sulfate, BaSO₄, indicates the presence of the sulfate ions in the sample. 3. Test for the Phosphate Add 1 M nitric acid, HNO₃, to 10 drops of sodium phosphate, Na₃PO₄, Ion. PO₄³ until the solution is acidic. (Test by dipping a stirring rod into your solution and touching the wet rod to a strip of blue litmus paper. The solution is acidic when the blue litmus changes to a red color). Then add 5 drops of 0.5 M ammonium molybdate, $(NH_4)_2$ MoO₄, and heat the test tube in a hot water bath. A powdery, light yellow precipitate indicates the presence of the phosphate ion. The molybdate solution is yellow. Be sure you see a precipitate before you conclude that phosphate ions are present. 4. Test for the Thiocyanate Add 10 drops of 3 M acetic acid, HC₂H₃O₂, to about 10 drops of 0.5 M Ion, SCN potassium thiocyanate, KSCN, and stir with a glass stirring rod. Add 1 or 2 drops of 0.1 M iron(III) nitrate, Fe(NO₃)₃. The formation of a deep red color indicates the presence of thiocyanate ions.

- 5. Test for Chloride Ion, Cl⁻
 A. Add 5 drops of 1 M nitric acid, HNO₃, to about 10 drops of 0.5 M sodium chloride, NaCl. Add 2 or 3 drops of 0.1 M silver nitrate, AgNO₃. A white precipitate of silver chloride, AgCl, confirms the presence of silver ions.
 - **B.** If thiocyanate ion is present, it will interfere with this test, since it also forms a white precipitate with silver nitrate. If the sample contains

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 SCN^- ion, put 10 drops of the solution into a medium-size test tube and add 10 drops of 1 M nitric acid. Boil the solution gently until the volume is reduced to half. This procedure will oxidize the thiocyanate and remove the interference. Then perform the chloride ion test as previously explained.

- 6. Test for the Aluminum lon, Al³⁺ Add 1 M ammonia, NH₃ dropwise to about 10 drops of 0.5 M aluminum chloride, AlCl₃, until the solution is basic (stir the solution with a glass stirring rod, then touch the wet rod to a strip of red litmus paper. When the solution is basic, the paper will turn blue). A white, gelatinous precipitate of aluminum hydroxide, Al(OH)₃, will form. Add 3 M acetic acid, HC₂H₃O₂, until the precipitate dissolves. Stir and add 2 drops of cathecol violet reagent. A blue solution indicates the presence of aluminum ions.
- **7. Test for the Copper(II)** Ion, Cu²⁺ Add concentrated ammonia, NH₃, dropwise to about 10 drops of 0.5 M copper(II) sulfate, CuSO₄. The development of a deep blue color indicates the presence of copper(II) ions.
- **8.** Analysis of an Unknown When you have completed all of the tests, obtain an unknown from your instructor. *Be sure to record the unknown number*. Analyze your solution by using 10-drop portions of the unknown and then applying the various tests to separate portions. Substitute the unknown for the compound that contains the ion you are testing for. For example, if you are testing for the CO_3^{2-} ion, do not add Na_2CO_3 ; use your unknown in its place. The unknown will contain more than one ion, so your test for a given ion may be affected by the presence of others (see Step 5B).

Name	Date	Section	

Experiment 13

Advance Study Assignment

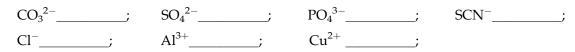
1. An unknown that might contain any of the seven ions studied in this experiment (but no other ions) has the following properties:

a. On addition of 1 M HCl, bubbles form.

b. When 0.1 M BaCl₂ is added to the acidified unknown, a white precipitate results.

c. When 0.1 M AgNO₃ is added to the unknown, a clear solution results.

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:



2. What is the difference between qualitative analysis and quantitative analysis?

Optional Assignment

Write net ionic equations for the reactions in this experiment in which the following ions are detected:

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Experiment 13

Work Page

Ions	Ions Observations Tested (Known)	Unknown	
Tested		Yes	No
CO3 ²⁻			
SO4 ²⁻			
PO4 ³⁻			
SCN^{-}			
Cl ⁻			
Al ³⁺			
Cu ²⁺			

Ions present _____

Name	Date	Section

Experiment 13

Report Sheet

_		Unknown	
Ions Tested	Observations (Known)	Yes	No
CO3 ²⁻			
SO4 ²⁻ PO4 ³⁻			
PO4 ³⁻			
SCN^{-}			
Cl ⁻			
Al^{3+}			
Cu ²⁺			

Ions present _____