Separation of Cations

Performance Goals

- **14–1** Prepare a solution containing five cations.
- 14–2 Separate and identify these cations using ion-specific reactions.
- 14–3 Analyze an unknown solution for certain cations.

CHEMICAL OVERVIEW

Qualitative analysis is a method that identifies the components of an unknown solution. In this experiment you will not only determine which ions are present, but you will also learn how to separate these ions in a mixture. This separation process is based on the specific chemical reactions each ion undergoes and also on the difference of solubility each compound has. You will use confirmatory tests also, to be sure that a particular ion is, in fact, present.

In this experiment you will learn to separate and identify five cations. The unknown solution may contain from one to five of the following ions:

$$Ag^{+} Pb^{2+} Hg_{2}^{2+} Fe^{3+} Ni^{2+}$$

First, you will mix five solutions, each containing one of the above cations, to give a "known" solution. Then, following the given procedure, you will separate and identify each cation. When you are finished, you will be given an unknown and asked to analyze that solution and determine which cations are present.

SAFETY PRECAUTIONS AND DISPOSAL METHODS

In some tests you will be required to use fairly concentrated acids and bases that are very corrosive. If these come in contact with skin, be sure to rinse the exposed area with plenty of cold water. Wear goggles while performing this experiment. You will also be using a hot-water bath. Be careful not to spill the hot water. Handle test tubes in the hot-water bath by using a test-tube holder.

Discard all solutions in a waste container.

PROCEDURE

- **A.** In a small beaker, mix 10 drops of each solution containing silver nitrate, AgNO₃, lead(II) nitrate, Pb(NO₃)₂, mercury(I) nitrate, Hg₂(NO₃)₂, iron(III) nitrate, Fe(NO)₃, and nickel(II) nitrate, Ni(NO₃)₂, to make a "known" solution. Stir well with a stirring rod.
- **B.** Start heating some water for a water bath.
- **C.** Pour about 1 mL of your known solution into a small test tube and add 10 drops of 6 M HCl. Mix thoroughly with a stirring rod (a small diameter rod works well in your small test tubes).
- **D.** Put the test tube into a centrifuge, making sure that there is another test tube containing about the same amount of liquid in the opposite opening. Start the centrifuge and let it spin for about 20–30 seconds.

The precipitate should be on the bottom of the test tube and the liquid layer should be fairly clear. If this is not the case, centrifuge the mixture for 10 seconds longer. The precipitate contains AgCl, Hg₂Cl₂, and PbCl₂.

Pour the liquid into a small, clean test tube and save it for further tests.

NOTE: You will be using several test tubes, containing solutions from different steps and also precipitates from separation steps. It is extremely important that you label these test tubes, designating the step from which they were obtained and the ions they contain.

- **E.** Add 1 to 2 mL of deionized water to the test tube containing the precipitate and stir it well with a glass rod. Centrifuge it for 20–30 seconds and discard the wash water.
- **F.** Add about 2 mL of deionized water to the precipitate and, using a testtube holder, place the test tube in a boiling water bath. (Be careful—the water level should be below the opening of the test tube!) Using a stirring rod, mix the contents of the test tube. Heat for about 3 minutes. The PbCl₂will dissolve, but not the AgCl or Hg₂Cl₂. Centrifuge the hot mixture and pour the hot liquid into another small test tube.
- **G.** Allow the solution from Step F to cool. Then, add one drop of 6 M acetic acid, $HC_2H_3O_2$, and a few drops of 0.1 M potassium chromate, K_2CrO_4 . If Pb^{2+} ions are present, $PbCrO_4$ will form, which is a bright yellow precipitate.
- **H.** To the precipitate from Step F, add 1 mL of 6 M ammonia, NH_3 , and stir thoroughly. Centrifuge the mixture and transfer the liquid into another test tube. If the remaining solid is gray or black, Hg_2^{2+} ions are present.
- **I.** To the liquid from Step H, add 6 M nitric acid, HNO₃, slowly until a blue litmus paper turns red. Do not dip the paper into the solution; instead touch the wet stirring rod to the strip. If Ag⁺ ions are present, the solution will turn cloudy, due to AgCl, which precipitates.
- **J.** To the solution from Step D, add 6 M NH₃ until it is basic (i.e., a red litmus paper turns blue). Add 1 mL more NH₃ and stir. Centrifuge the mixture. The brown precipitate is Fe(OH)₃ and the nickel is in solution as a complex ion. Pour the solution into another test tube.

- **K.** Dissolve the precipitate from Step J in about 0.5 mL of 6 M hydrochloric acid, HCl, and add 2 mL of deionized water. Stir, then add 2 drops of 0.5 M potassium thiocyanate, KSCN. If Fe³⁺ ions are present, a deep red solution will form.
- **L.** To the solution from Step J, add a few drops of dimethylglyoxime reagent and stir. If Ni²⁺ ions are present, a rose-colored precipitate will form.
- **M.** Obtain an unknown. Be sure to record the unknown number! The unknown may contain from one to five cations.

Perform Steps C through L exactly as you did for the known, except use the unknown where you used the known solution before. If, at some point, due to the absence of an ion, you do not get a precipitate or color change, proceed to the next step (i.e., if there is no precipitate, there is no need to centrifuge!).

Record your observations and list the ions present in your unknown.

Name	Date	Section

Experiment 14

Advance Study Assignment

1. A solution contains Ag⁺ ions and Pb²⁺ ions. Describe how you would separate them and what tests you would perform to verify that the specific ion is present.

- **2.** An unknown that might contain any of the five cations studied in this experiment (but no other ions) has the following properties:
 - **a.** On addition of HCl a white precipitate forms.
 - **b.** The white precipitate is insoluble in hot water.
 - c. After centrifuging the precipitate, the solution yields a brown precipitate when NH₃ is added.

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:

 Ag^+ _____; Pb^{2+} _____; Hg_2^{2+} _____; Fe^{3+} _____; Ni^{2+} _____

Name	Date	Section

Experiment 14

Work Page

	Determining Reaction	Unknown	
Ions Tested	(Known)	Yes	No
Ag^+			
Pb ²⁺			
Hg_2^{2+}			
Fe ³⁺			
Ni ²⁺			

Unknown number_____. Ions present _____

Name	Date	Section

Experiment 14

Report Sheet

	Determining Reaction	Unknown	
Ions Tested	(Known)	Yes	No
Ag ⁺			
Pb ²⁺			
$\mathrm{Hg_2}^{2+}$			
Fe ³⁺			
Ni ²⁺			

Unknown number_____. Ions present_