Investigation 3: Preparation and Analysis of a Compound

Focus Questions: How can we determine the empirical formula of the potassium iron oxalate compound synthesized in lab? What is the percent yield of the synthesis reaction?

Background

Synthesizing compounds in the laboratory – especially those that are new, medically useful, or brightly colored – is an exciting facet of chemistry. However, determining the chemical formula of the compound produced can be a challenge. In this series of investigations, you will synthesize a complex compound and then use several techniques to determine the empirical formula of the compound.

Overview

The compound that will be synthesized has the general formula $K_aFe_b(C_2O_4)_c \cdot dH_2O$, where $a$, $b$, $c$, and $d$, are integers that give the relative molar quantities of each component in the compound. The synthesized compound will be isolated by filtration and then analyzed for percent potassium, percent oxalate, and percent water, all over a period of several weeks. The percent iron will be found by difference once the percentages of the other three components are known. These percentages will then be used to calculate the empirical formula of the synthesized compound.

Synthesis and Isolation of the Compound

Preparation of NaOH Solution, Dehydration and Analysis for Potassium

Pre-lab required reading


Technical Primers:

- Keeping a Laboratory Notebook
- Volumetric glassware use – General
- Vacuum filtration
- Volumetric glassware use - Buret
- Volumetric glassware use – volumetric flask
- Volumetric glassware use – volumetric pipet
- Spectrophotometry
Using the spectrophotometer

Safety and Waste Disposal

- Eye protection should be worn at all times.
- Solutions should be stirred frequently to prevent bumping while being heated.
- Waste solids should be placed in designated container.
- Methanol is flammable and should not be used near sparks or flames.
- Dispose of waste methanol in a volatile liquids container.
- Acetone is flammable and should not be used near sparks or flames.
- Dispose of waste acetone in a volatile liquids container.
- Sodium hydroxide and hydrochloric acid solutions are corrosive. Wear goggles at all times. Small amounts of base or acid on the skin should be rinsed off with water at any sink. Spills should be wiped up immediately. Large amounts of base or acid on the skin should be rinsed off at once in the safety shower.
- Dispose of all waste materials in the appropriate designated container.

Terminology

- **Elute**: to separate one material from another, usually by means of a solvent
- **Eluent**: a substance used as a solvent in separating a mixture

Procedures

**Synthesis of the compound**

In a 100- or 150-mL beaker, prepare a solution consisting of about 5.3 g (measure and record the precise mass) FeCl$_3$·6H$_2$O in 8 mL of DI water. In a second 100- or 150-mL beaker, combine 12 g of K$_2$C$_2$O$_4$·H$_2$O and 20 mL of DI water. Heat this second solution, with stirring, until all the potassium oxalate is dissolved. Pour the iron(III) chloride solution into this second, hot solution and stir. Cover the beaker with a watch glass and place it in a location where it will be undisturbed for at least 24 hours or cool with an ice bath and scratch the bottom inside of the beaker with a stirring rod.

**Filtration and mass of the compound**

Obtain about 10 mL of chilled methanol in a small beaker and place the beaker in an ice bath. Assemble an aspiration filtration apparatus as instructed. Vacuum filter the crystals, rinsing them twice with small amounts of cold methanol. Spread the crystals on a watch glass and leave them in a safe place to air dry for about 1 hour. Measure the mass of the dried, synthesized compound. Crush the solid with a mortar and pestle for use during the analysis portion of the investigation.

**Preparation of NaOH solution (to be used in the analysis of potassium ion)**

Calculate the volume of 50.5% NaOH$_{aq}$ (density = 1.54 g/mL) required to prepare 250 mL of 0.1 M NaOH$_{aq}$. Place about 200 mL of water in a clean beaker and add the calculated volume of 50.5% NaOH$_{aq}$. Add more water to bring the final volume of the solution to 250 mL. Stir thoroughly. Combine your solution with the sodium hydroxide solutions made by the other members of the lab. Note: The precise concentration of this combined solution will be determined later (Standardization of NaOH).

**Dehydration (analysis for water)**

Weigh a dried weighing bottle. Place about 1 g of the synthesized compound in the weighing bottle and measure and record the precise mass of the weighing bottle and sample. Place the weighing bottle into a small, labeled beaker (to make the bottle easier to transport) and place the beaker in an oven at 110 °C for at least one hour. Cool the weighing bottle containing the dehydrated sample in a desiccator, then record the final precise mass of the sample and bottle.
**Standardization of NaOH\(_{aq}\)**

Place 0.4 – 0.5 g of dried potassium hydrogen phthalate (be sure to record the precise mass) into a clean flask and add about 50 mL of water to dissolve the solid. Add 3 – 4 drops of phenolphthalein indicator solution to the flask. Prepare a buret containing the 0.1 M sodium hydroxide solution prepared by the class in week two. Titrate the potassium hydrogen phthalate with the sodium hydroxide solution and report the mass of potassium hydrogen phthalate used as well as the volume of sodium hydroxide solution required to your instructor.

**Ion Exchange (analysis for potassium ion)**

Obtain an ion exchange column. **Note that the resin in the column has been kept wet – it is essential for proper ion exchange that the resin never be allowed to become dry.** Add about 5 mL of water to the column and test the eluent with litmus paper to be sure that it is not acidic. If the eluent is acidic, continue to add 5 mL aliquots of water until the eluent is no longer acidic.

Dissolve 0.3 – 0.5 g of the original synthesized compound in a minimum amount of water (start with about 20 mL) in a beaker – the mixture may need to be warmed slightly in order to completely dissolve the solid, but do not exceed a temperature that is comfortable to the touch. Slowly add this solution to the top of the ion exchange column. As the liquid level falls and approaches the top of the resin, add a small amount of water to keep the liquid level above the resin. Continue to add water until the yellow-green compound solution has completely eluted from the column. Add an additional 5 mL of water to the column. Give the solution to your partner to titrate (see below). Place a second beaker under the ion exchange column. Add about 1 - 2 mL of 6 M HCl\(_{aq}\) to the column and then add portions of water to move the acid through the column. Flush the column with water until the eluent is no longer acidic. Cap the bottom of the column and check to see that there is about 1 inch of water standing above the resin level. Cover the top of the column.

Tritrate the flask containing the eluted compound solution with sodium hydroxide solution to a phenolphthalein endpoint. The endpoint will appear amber rather than pink as in the standardization titration.

**Reaction with a standard Cerium solution (analysis for oxalate)**

Dissolve about 0.1 g of the original synthesized compound (measure and record the precise mass) in 1M H\(_2\)SO\(_4\)\(_{aq}\) in 50-mL volumetric flask. Clean and dry two cuvettes. Fill one cuvette with 1M H\(_2\)SO\(_4\)\(_{aq}\) and use this solution to calibrate the spectrophotometer. Using a volumetric pipet, transfer 1.00 mL of the prepared solution of synthesized compound into the second cuvette. Record the absorbance of the solutions at 450nm. Using a micropipette, transfer 60\(\mu\)L of 0.05000 M Ce\(^{4+}\) (prepared using 1M H\(_2\)SO\(_4\)\(_{aq}\) as solvent) to the cuvette containing the solution of synthesized compound and record the absorbance. Continue adding 60\(\mu\)L aliquots of 0.05000 M Ce\(^{4+}\) and recording the absorbance of the resulting solutions until at least two aliquots have been added after the minimum recorded absorbance.

**Preparation of standardized Ce\(^{4+}\) Solution (analysis of oxalate)**

Prepare 25.0 mL of 0.05000 M Ce\(^{4+}\) solution volumetrically using (NH\(_4\))\(_2\)Ce(NO\(_3\))\(_6\) and 1 M H\(_2\)SO\(_4\)\(_{aq}\) as the solvent. Store the solution in a clean, dry, labeled bottle.

**References**

