Investigation 5: Preparation and Analysis of a Compound

Focus Questions: *What is the percent yield of a potassium iron oxalate compound synthesized in lab? What strategies and techniques are used to determine its empirical formula?*

Required prelab preparation

Review Sections 7.3-7.5 and 8.6-8.7 in *Chemistry: an Atoms-focused Approach (4th ed)* and the following technical primers: keeping a laboratory notebook | general use of volumetric glassware | vacuum filtration | use of a buret | use of a volumetric flask | use of a volumetric pipet | spectrophotometry

Background and overview

Chemical synthesis is an exciting field—it is an engine driving discovery of new compounds that find use as catalysts, pharmaceuticals, colorants, preservatives, and structural materials. Equally important is the field of analytical chemistry, with which the identity and composition of a compound can be found, and which is central to many important applications (quality assurance of drugs and consumer goods, detection of environmental contaminants, etc.). This investigation combines aspects of both synthetic and analytical chemistry by creating and isolating an inorganic complex and then determining its exact composition, or empirical formula.

The compound to be synthesized has the general formula $K_aFe_b(C_2O_4)_c \cdot dH_2O$, where a, b, c, and d are integers representing the relative molar quantities of potassium, iron, oxalate, and water, respectively. Over the course of three weeks, the compound is formed by combining aqueous solutions of potassium oxalate hydrate and iron(III) chloride hexahydrate, isolated by filtration, and then analyzed for each component. Figure 1 provides an overview of the different analytical approaches employed. Once the weight percent of each component is known, the empirical formula can be determined using the conventional method employed for combustion (or C,H,N) analysis.

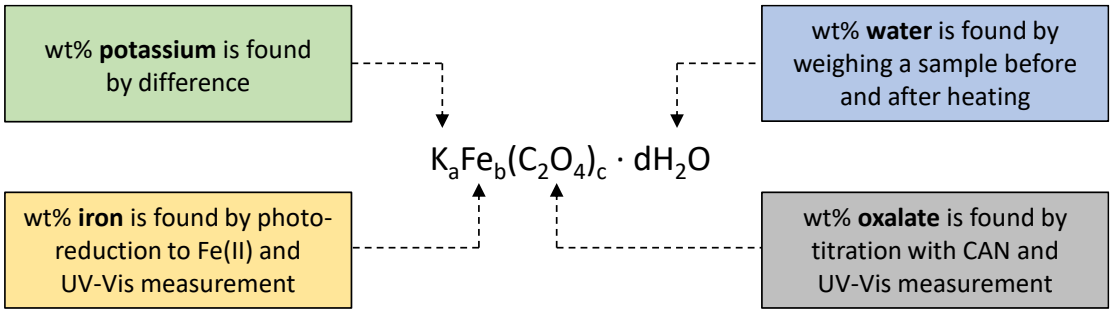

Figure 1. Analytical strategy for finding the weight percent of each component in the synthesized compound.

Procedures

Week 1

Synthesis and isolation. In a 100 mL beaker, dissolve about 5.3 g (record precise mass) iron(III) chloride hexahydrate in 8 mL deionized water. In a second 100 or 150 mL beaker, dissolve about 12 g (record precise mass) potassium oxalate hydrate in 20 mL deionized water, heating and swirling until all the solid is dissolved. Pour the iron(III) chloride solution into the hot potassium oxalate solution, rinse forward with about 1 mL deionized water, and swirl to combine. Cover the beaker with a watch glass and set aside in the dark for 1 h to cool and crystallize. One may use an ice bath and scratch the bottom inside of the beaker with a stirring rod to promote crystallization, if needed.

Yield determination. When crystallization is complete, chill 10 mL methanol in a small beaker on an ice bath. Assemble the vacuum filtration apparatus as instructed to collect the crystals by filtration. Once all the crystals are in the filter funnel, wash them with the cold methanol in two portions. Let the vacuum run for another minute or so, then transfer the crystals into a labeled, previously weighed watch glass. Spread the crystals on the watch glass and leave them in a safe place to air dry for about 1 hour. Measure the mass of the dried, synthesized compound on the watch glass. Subtract the weight of the watch glass to find the net weight of the dried compound.

Crush the solid with a mortar and pestle to a fine powder and transfer to a labeled sample vial for use during the analysis portion of the investigation. Store in the dark when not in use. Once the formula of the compound is determined, this mass will be used to calculate percent yield.

Week 2

Water analysis. Weigh a dried sample vial. Place about 1 g of the synthesized compound in the vial 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 identify) 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.

Preparation of 0.05000 M Ce^{4+} solution. To a 10 mL volumetric flask, add the appropriate amount (calculated in the prelab assignment or class) of ceric ammonium nitrate (CAN). Add about 7 mL of 1 M aqueous sulfuric acid and swirl to solution. Dilute to the mark with 1 M aqueous sulfuric acid, stopper, and invert several times to mix. Transfer to a clean, dry, labeled bottle for storage.

Preparation of compound solution for oxalate and iron analysis. In a 50 mL volumetric flask, dissolve about 0.2 g (record precise amount) of original (non-dehydrated) compound in 1 M aqueous sulfuric acid. Swirl to completely dissolve then fill the volumetric flask to the mark with 1 M sulfuric acid.

Oxalate analysis. Clean and dry two cuvettes. Fill one cuvette with 1M aqueous sulfuric acid to calibrate the spectrophotometer. Using a micropipette, transfer 1,000 µL of the compound solution to a second cuvette and measure the absorbance at 450 nm. Using a micropipette, add 100 µL of 0.05000 M Ce^{4+} solution to the cuvette, carefully mix with a plastic pipette, and measure the absorbance at 450 nm. Continue this process of adding 100 µL aliquots until the absorbance reaches a minimum and at least three increasing data points are recorded.

Iron analysis. Add 5 mL of compound solution (prepared above) to a 7 mL sample vial and place in the center of a 450 nm photoreactor. Place the cover on the reactor and irradiate for 2 min. To a 10 mL volumetric flask containing about 5 mL deionized water, add 100 µL of the irradiated sample, 1,000 µL ammonium acetate buffer, and 1,000 µL developing solution. Dilute to the mark with deionized water, invert to mix, and let stand 5 min to develop. Measure the absorbance at 510 nm. Use the Beer's law plot below to determine the concentration of iron in the sample.

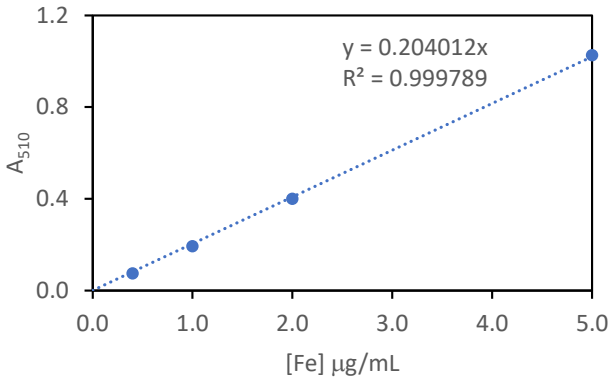

Figure 4. Beer's Law plot for the iron(II) phenanthroline complex, in which absorbance at 510 nm is correlated to the iron concentration of the analyzed solution (as µg Fe per mL solution).

Safety and Waste Disposal

- Eye protection must be worn at all times.
- Heated solutions should be constantly agitated to prevent bumping
- Potassium oxalate monohydrate is harmful if swallowed or in contact with skin, causes serious eye irritation, and is harmful to aquatic life.
- Iron(III) chloride hexahydrate is harmful if swallowed, causes skin irritation and serious eye damage, and is toxic to aquatic life.
- Methanol is highly flammable, toxic if swallowed, in contact with skin or if inhaled, and causes damage to organs (eyes, central nervous system).
- 1M Sulfuric acid may be corrosive to metals, and causes skin irritation and serious eye irritation.
- Ceric ammonium nitrate is an oxidizer and may intensify fire, may be corrosive to metals, is harmful if swallowed, causes severe skin burns and eye damage, may cause an allergic skin reaction, and is very toxic to aquatic life with long lasting effects.
- Dispose of methanol in the volatile liquids waste container.
- Dispose of waste solids in the designated container.
- Small amounts of hydrochloric and sulfuric acid may be rinsed down the drain with plenty of water.

References

Atkins, P.; Jones, L. “Chemical Principles: The Quest for Insight”, 5th ed.; Freeman: New York. 2010.

Johnson, R. C. *J. Chem. Educ*. **1970**, *47*, 702.

Olmsted. J. *J. Chem. Educ*. **1984**, *61*, 1098-9.