Experiment 5
Synthesis and Analysis of a Really Weird and Curiously Green Iron Compound

Part 1: Synthesis

CH 204 Fall 2006

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Last Week

Acid/Base titration

Standardizing a solution

moles H^+ = moles OH^-

Calculating moles by $\frac{\text{grams}}{\text{MW}}$ and Molarity x Volume

Three-week experimental adventure quest!

This week: Synthesis of a potassium oxalatoferrate salt.

$K_x[Fe_y(C_2O_4)_x]\cdot zH_2O$

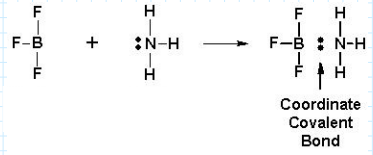
Starting material $\xrightarrow{\text{Series of reactions}}$ Product

"Precursors", "Intermediate products"

Next two weeks: Qualitative identification of the compound through quantitative analysis of oxalate and iron.

Coordinate Covalent Bonds

Coordinate covalent bond: two shared electrons in a bond, but *both electrons come from the same atom.*



Our compound will have coordinate covalent bonds between the central iron ion and the oxygen atoms in oxalate.

What is potassium oxalatoferrate?

Oxa-who?

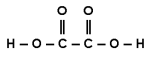
An ionic crystal with a big, covalently-bound anion.

$K_x[Fe_y(C_2O_4)_x]\cdot zH_2O$

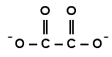
Cation: K^+

Anion: $Fe_y(C_2O_4)_x^{x-}$

Waters of hydration



Oxalic acid



Oxalate ion

Procedure Overview

- Dissolve an Fe^{2+} salt in water and add oxalate to precipitate the iron as Iron (II) Oxalate solid.
- Oxidize the iron to Fe^{3+} in the presence of excess oxalate. The precipitate will dissolve as the complex ion forms in solution.
- Precipitate the iron complex ion as the potassium salt by adding ethanol to the mix.

A sneak peek at redox half-reactions

Oxidation half reaction:

$$Fe^{2+} \longrightarrow Fe^{3+} + e^-$$

Reduction half reaction:

$$H_2O_2 + 2\,H^+ + 2\,e^- \longrightarrow 2\,H_2O$$

Add the two halves:

$$2\,Fe^{2+} + H_2O_2 + 2\,H^+ \longrightarrow Fe^{3+} + 2\,H_2O$$

Grading this lab

- No real data to speak of, so not the usual lab report
- Discussion questions count for more
- Record your observations during the experiment – precipitation, color changes, evolution of gases, dissolving of precipitates.

WARNING!

Follow lab directions carefully or there will be no green crystalline delight for you!

(And this will make you cry.)

Do NOT overheat solutions in the lab today!

Potassium oxalate ≠ Oxalic acid!

If crystals don't form in the end, add another 10 ml of ice-cold ethanol.

What's going to be on the quiz next week?

Look at the post-lab problems. Be able to calculate:

Limiting reagent

Theoretical yield

Percent yield