

Experiment 5

Synthesis und Analysis of a Complex Iron Compound

Part 1: Synthesis

CH 204 Spring 2007

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Last Week

Standardizing a solution

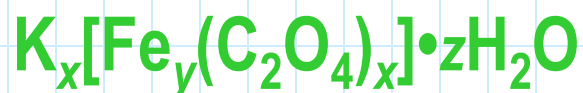
Acid/Base titration



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

Three-week experimental adventure quest!

This week: **Synthesis of a potassium oxalatoferrate salt.**



Series of reactions

Starting material \longrightarrow \longrightarrow \longrightarrow \longrightarrow Product

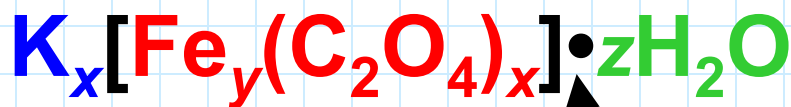
"Precursors", "Intermediate products"

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

What is potassium oxalatoferrate?

Oxa-who?

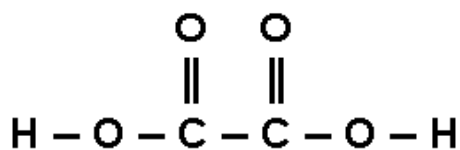
An ionic crystal with a big, covalently-bound anion.



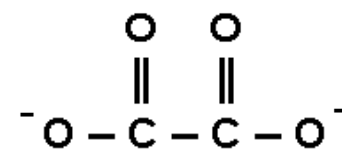
Cation: K^+

Anion: $\text{Fe}_y(\text{C}_2\text{O}_4)_x^{x-}$

Waters of hydration



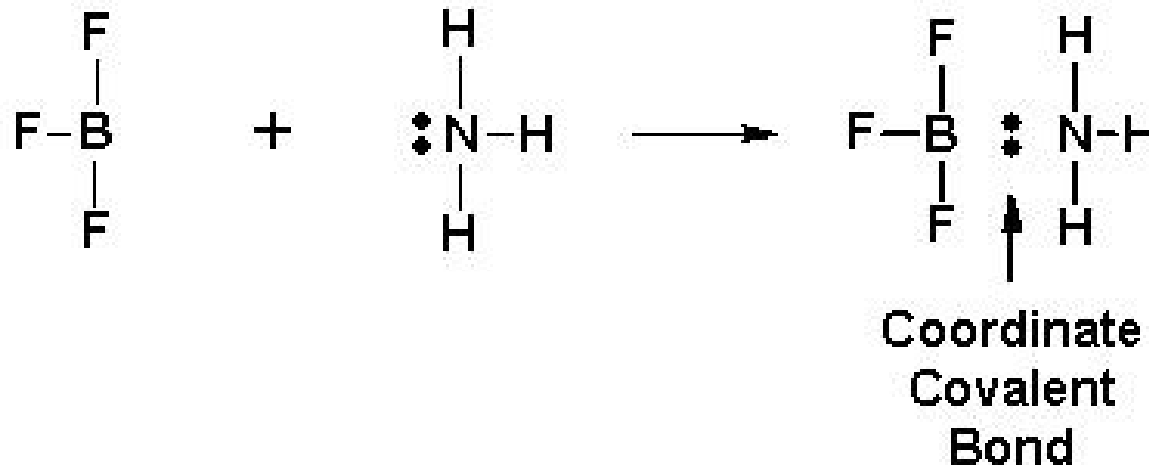
Oxalic acid



Oxalate ion

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⁺³ ion and the oxygen atoms in oxalate.

Procedure Overview

- Dissolve an Fe^{2+} salt in water and add oxalic acid to precipitate the iron as a yellow solid, Iron (II) Oxalate. (Steps 1-8)
- Oxidize the iron to Fe^{3+} in the presence of excess oxalate. The precipitate will dissolve as the complex ion forms in solution. (Steps 9 – 12)
- Precipitate the iron complex ion as the potassium salt by adding ethanol to the mix. (Steps 13 – 15)

WARNING!

Follow lab directions carefully or there will be
no sparkly green crystalline delight for you!
(And this will make you cry.)

Do NOT overheat solutions in the lab today!

Potassium oxalate \neq Oxalic acid!

If crystals don't form in the end, slowly add up
to 10 ml more of ice-cold ethanol.

Grading this lab

- No real data to speak of, so not the usual lab report
- Record your observations during the experiment — precipitation, color changes, evolution of gases, dissolving of precipitates. You will be graded on these!
- Discussion questions count for more points this time

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