

# Experiment 5

## Synthesis und Analysis uff ein Complex Iron Compound

### Part 1: Synthesis

CH 204 Spring 2008

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# Last Week

## Standardizing a solution

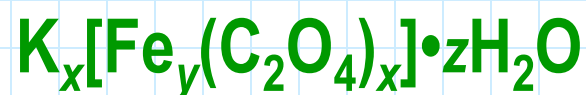
### Acid/Base titration



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

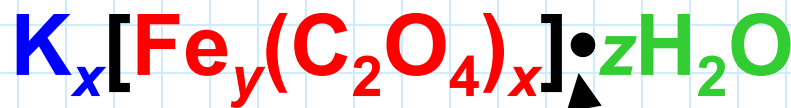
"Precursors", "Intermediate products"

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

# Was ist potassium oxalatoferrate?

Oxa-who?

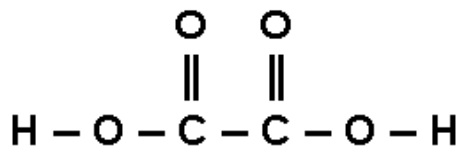
An ionic crystal with a big, covalently-bound anion.



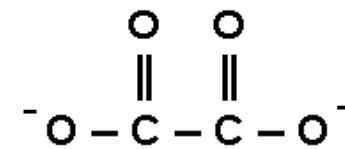
Cation:  $\text{K}^+$

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

Waters of hydration



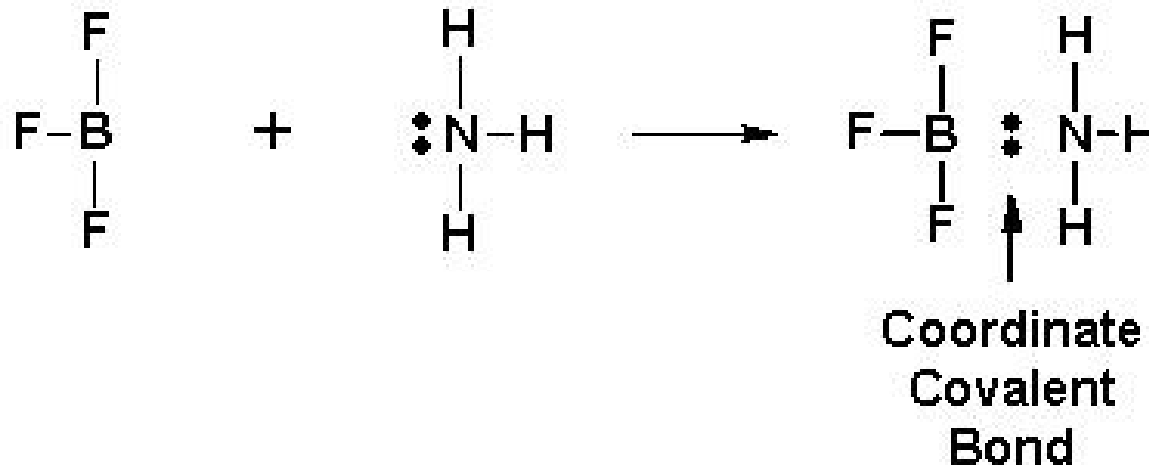
Oxalic acid



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<sup>+3</sup> ion and the oxygen atoms in oxalate.

# Procedure Overview

- Dissolve an  $\text{Fe}^{2+}$  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  $\text{Fe}^{3+}$  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 a green crystal 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

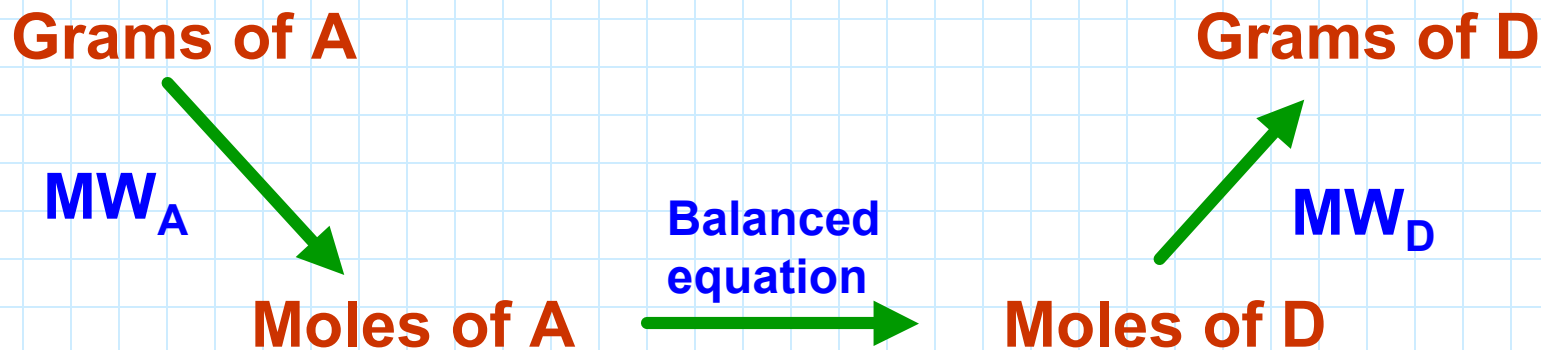
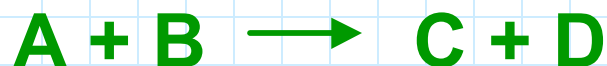
- No real data to speak of, so not the usual lab report
- **Record your observations** during the experiment — precipitation, color changes, evolution of gases, dissolving of precipitates. You will be graded on these!
- Discussion questions count for more points this time



# Post-lab 5 overview

Theoretical yield and limiting reagent problems typically follow the same three-step procedure:

You are given the number of grams of a reactant (A), and are asked for the number of grams of a product (D).



# Pre-lab 6 preview

Question 5: Determining oxidation states.

Not there yet in 302, and it's not in the lab manual.

Look at the help sheet on the Freebies page of the class web site:

<http://courses.cm.utexas.edu/banderson/ch204/freebies.html>

# Final Exam Part 4

There are 9 parts total, and we count the best 8.

After today you are almost halfway done with the final exam.

The next few quizzes will always have at least one question similar to the post-lab questions. Make sure you understand how to do the post-labs!