Experiment 5
Synthesis und Analysis of ein
Complex Iron Compound

Part 1: Synthesis

CH 204    Spring 2008
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Last Week
Standardizing a solution
Acid/Base titration
moles H⁺ = moles OH⁻
moles acid x " of H⁺ = moles base x " of OH⁻
Calculating moles by grams and Molarity x Volume
MW

Three-week experimental adventure quest!
This week: Synthesis of a potassium oxalatoferrate salt.
K₃[Fe₃(C₂O₄)₆]·2H₂O
Series of reactions
Starting material → → → → Product
"Precursors", "Intermediate products"
Next two weeks: Qualitative identification of the compound through quantitative analysis of oxalate and iron.
Was ist potassium oxalatoferrate?

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

\[ \text{K}_x[\text{Fe}_y(\text{C}_2\text{O}_4)_z] \cdot z\text{H}_2\text{O} \]

Cation: K⁺

Anion: Fe₇(\text{C}_2\text{O}_4)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⁺⁺⁺ ion and the oxygen atoms in oxalate.

Procedure Overview

• Dissolve an Fe²⁺ salt in water and add oxalic acid to precipitate the iron as a yellow solid, Iron (II) Oxalate. (Days 1–6)

• Oxidize the iron to Fe³⁺ in the presence of excess oxalate. The precipitate will dissolve as the complex ion forms in solution. (Days 9 – 12)

• Precipitate the iron complex ion as a green crystal by adding ethanol to the mix. (Days 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 ≠ Oxalic acid!

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

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**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

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**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).

\[
A + B \rightarrow C + D
\]

<table>
<thead>
<tr>
<th>Grams of A</th>
<th>Grams of D</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW_A</td>
<td>MW_D</td>
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Moles of A — Balanced equation — Moles of D

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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!