# Experiment 6

## Synthesis und Analysis of ein Magical Green Crystal

Part Deux: Oxalate Content Analysis by Redox Titration Using a Vile Purple Fluid

CH 204 Spring 2009

Dr. Brian Anderson

## But first...

Last week:

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

Coordinate covalent bonds and metal complex ions

Calculating limiting reagent, theoretical yield, and percent yield

This week:

Oxidation-Reduction (Redox) chemistry

## What is redox chemistry?

Moving electrons between different atoms:

$$Cu^{2+}_{(aq)} + Zn_{(s)} \longrightarrow Cu_{(s)} + Zn^{2+}_{(aq)}$$

$$Cu^{2+}_{(aq)} + 2\ e^- \longrightarrow Cu_{(s)}$$

$Cu^{2+}$ gains electrons.
$Cu^{2+}$ is REDUCED.

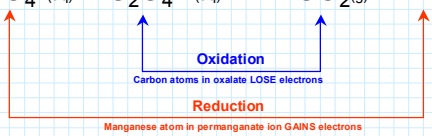
$$Zn_{(s)} \longrightarrow 2\ e^- + Zn^{2+}_{(aq)}$$

$Zn_{(s)}$ loses electrons.
$Zn_{(s)}$ is OXIDIZED.

## Our redox reaction

We will use $MnO_4^-$ to oxidize the oxalate ligands surrounding the $Fe^{3+}$. The carbon in the oxalate ions will be oxidized, and the oxalate will change from $C_2O_4^{2-}$ to $CO_{2(g)}$.

$$MnO_4^-{}_{(aq)} + C_2O_4^{2-}{}_{(aq)} \longrightarrow CO_{2(g)} + Mn^{2+}{}_{(aq)}$$




Hey! This reaction is not balanced!

## Balancing redox reactions

Separate the overall equation into two half-reactions. For each half-reaction:

1. Balance the main atom.
2. Add $H_2O$ to balance O.
3. Add $H^+$ to balance H.
4. Balance the charge using electrons.
5. Equalize electrons between the half-reactions.

When you're done, add the two half-reactions and cancel out the electrons.

Let's try a few. To the Doc Cam!

## Oxidation half-reaction

Here's our overall reaction again:

$$MnO_4^-{}_{(aq)} + C_2O_4^{2-}{}_{(aq)} \longrightarrow CO_{2(g)} + Mn^{2+}{}_{(aq)}$$

Oxidation of $C_2O_4^{2-}$ to $CO_2$ is simple enough:

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-$$

## Reduction half-reaction

Overall reaction:

$$MnO_4^-(aq) + C_2O_4^{2-}(aq) \longrightarrow CO_2(g) + Mn^{2+}(aq)$$

The oxidizing agent, $MnO_4^-$, gets reduced to $Mn^{2+}$

$$MnO_4^- \rightarrow Mn^{2+} + ???$$

Balance Mn
Balance O using $H_2O$
Balance H using $H^+$
Balance charge using $e^-$

## Reduction half-reaction solved!

$$MnO_4^- + 8\,H^+ + 5\,e^- \rightarrow Mn^{2+} + 4\,H_2O$$

Balance Mn
Balance O using $H_2O$
Balance H using $H^+$
Balance charge using $e^-$

## Add the two half reactions

First multiply the equations in order to equalize the electrons between the two half-reactions:

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^- \quad \times 5$$
$$8H^+ + MnO_4^- + 5e^- \rightarrow Mn^{2+} + 4H_2O \quad \times 2$$

$$5C_2O_4^{2-} \rightarrow 10CO_2 + 10e^-$$
$$16H^+ + 2MnO_4^- + 10e^- \rightarrow 2Mn^{2+} + 8H_2O$$

Es gibt keine Elektronen, ja?
They all cancel out!
Sehr gut?
Ja, sehr gut!

The equation for the overall reaction is:

$$16H^+ + 2MnO_4^- + 5C_2O_4^{2-} \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$$

## Always balance in acidic solution

Balancing redox half-reactions is as easy as 1-2-4.

1) Balance the main atoms
2) Balance oxygens using $H_2O$
3) Balance hydrogens using $H^+$
4) Balance charge using $e^-$

## What if the solution is basic?

Always balance the equation in acidic solution,
and if it's supposed to be in basic solution,
just add one $OH^-$ to both sides
For each $H^+$ in the reaction.

Just like this...

Permanganate is reduced to manganese (IV) oxide in basic solution:

$$MnO_4^- \rightarrow MnO_2$$

Balance O using $H_2O$: $MnO_4^- \rightarrow MnO_2 + \mathbf{2H_2O}$

Balance H using $H^+$: $MnO_4^- + \mathbf{4H^+} \rightarrow MnO_2 + 2H_2O$

Balance charge using $e^-$: $MnO_4^- + \mathbf{3e^-} + 4H^+ \rightarrow MnO_2 + 2H_2O$

We got 4 $H^+$, so add 4 $OH^-$ to both sides!

$MnO_4^- + 3e^- + 4H^+ + \mathbf{4OH^-} \rightarrow MnO_2 + 2H_2O + \mathbf{4OH^-}$

4 $H^+$ + 4 $OH^-$ → 4 $H_2O$, so delete spectator water molecules:

$MnO_4^- + 3e^- + 2H_2O \rightarrow MnO_2 + 4OH^-$

## Balancing redox reactions review

- Separate the reactants into half reactions.
- Balance the main atom.
- Balance the half-reactions using $H_2O$ to balance O, then use $H^+$ to balance H. Balance the charge with electrons.
- Equalize electrons and add the two half-reactions — electrons must cancel.
- If necessary, convert acidic solution to basic by adding $OH^-$ to both sides and crossing out spectator water molecules.

## Today: Sample prep and three titrations

Land mine! 1:1 mixture of ethanol/water means mix them together in a beaker BEFORE you pour them in!

The $KMnO_4$ solution is already standardized and ready to go.

Make sure you record the concentration: 0.0372 M.

Take only about 50 ml of $KMnO_4$

## Valuable time-saving tips!

Step 10: Start titrating while the sample is heating -- don't wait for 70°

Step 12: You are waiting two minutes for the purple color to go away. As soon as it goes away, start titrating again.

Step 13: In the titration, the solution starts out yellow and fades to colorless before it reaching the endpoint. As long as it's still yellow, you still have a ways to go.

## After Spring Break

Experiment 7 is broken into two weeks.

First we will do parts 2 and 3.

The following week we will do parts 1 and 4.

I will post more details on the class web page after Experiment 6.

## Quiz Time

Mini-Final part 5 of 9 – we're past the halfway mark.

Dr. Anderson – NO OFFICE HOURS THIS WEEK.

After spring break – Quiz 6. Balancing redox reactions (like post-lab problems 1, 2, and 3), and a redox reaction stoichiometry problem (like post-lab problems 4 and 5).