

Experiment 6

Synthesis und Analysis uff ein Magical Green Crystal

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

CH 204 Fall 2007

Dr. Brian Anderson

But first...

Last week:

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

Metal complex coordination compounds

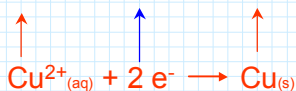
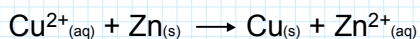
Calculating limiting reagent, theoretical yield,
and percent yield

This week:

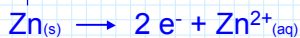
Oxidation-Reduction (Redox) chemistry

What is redox chemistry?

Moving electrons between different atoms:



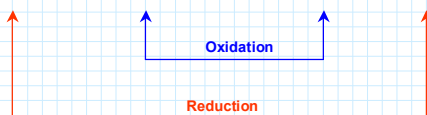
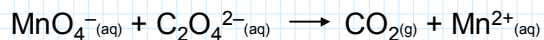
Cu^{2+} gains electrons.
 Cu^{2+} is REDUCED.



$Zn_{(s)}$ 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 $\text{C}_2\text{O}_4^{2-}$ to $\text{CO}_2(\text{g})$.



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.

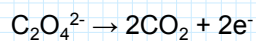
When you're done, add the two half-reactions and cancel out the electrons.

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

Oxidation half-reaction

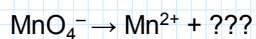
Oxidation of $\text{C}_2\text{O}_4^{2-}$ to CO_2 is simple enough:

(Remember, half-reactions do not include the other reactant)



Reduction half-reaction

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



Balance Mn

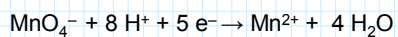
Balance O using H_2O

Balance H using H^+

Balance charge using e^-

Reduction half-reaction solved!

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



Balance Mn

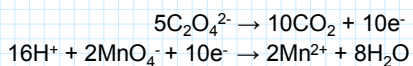
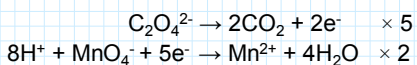
Balance O using H_2O

Balance H using H^+

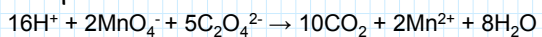
Balance charge using e^-

Add the two half reactions

First multiply the equations in order to balance out the electrons:



The equation for the overall reaction is:



Always balance in acidic solution

As easy as 1-2-4.

- 1) Balance the oxidized/reduced atoms
- 2) Balance oxygens using H_2O
- 3) Balance hydrogens using H^+
- 4) Balance charge using e^-

What if the solution is basic?

Here's what Whitten, Davis, Peck, and Stanley say:

To balance in a basic solution, for each O needed

(1) Add *two* OH^- to the side needing O

and

(2) Add *one* H_2O to the other side

Then, for each H needed,

(1) Add *one* H_2O to the side needing H

and

(2) Add *one* OH^- to the other side.

Here's what the current book suggests

"In basic solution, balance O by using H_2O ; then balance H by adding H_2O to the side of each half reaction that needs H and adding OH^- to the other side.

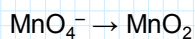
"When we add $\dots \text{OH}^- \dots \rightarrow \dots \text{H}_2\text{O} \dots$ to a half-reaction, we are effectively adding one H atom to the right. When we add $\dots \text{H}_2\text{O} \dots \rightarrow \dots \text{OH}^- \dots$ We are effectively adding one H atom to the left. Note that one H_2O molecule is added for each H atom needed."

Do it the E-Z way instead

Balance the equation in acidic solution,
and if it's supposed to be in basic solution,
just add enough OH^- to both sides
to get rid of all the H^+ .

Just like this...

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

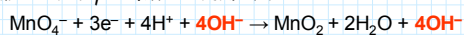


Balance O using H_2O : $\text{MnO}_4^- \rightarrow \text{MnO}_2 + 2\text{H}_2\text{O}$

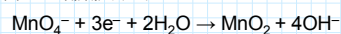
Balance H using H^+ : $\text{MnO}_4^- + 4\text{H}^+ \rightarrow \text{MnO}_2 + 2\text{H}_2\text{O}$

Balance charge using e^- : $\text{MnO}_4^- + 3e^- + 4\text{H}^+ \rightarrow \text{MnO}_2 + 2\text{H}_2\text{O}$

Add one OH^- for every H^+ . Add OH^- to both sides!



Combine waters and delete redundant waters:



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.
- 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 **KMnO₄** solution is already standardized and ready to go. Make sure you record the concentration: **0.0369 M**.

Take no more than **60 ml** of **KMnO₄**

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

Back to College Station

I will be gone again this week from **Wednesday** through **Friday**.

I will not have office hours on **Wednesday**.

You can go to **Dr. Leytner's** office hours on **Thursday** from **4:00** to **5:00**

I will have e-mail access in the evenings.
