

# 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

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# But first...

Last week:

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

Coordinate covalent bonds and metal complex ions

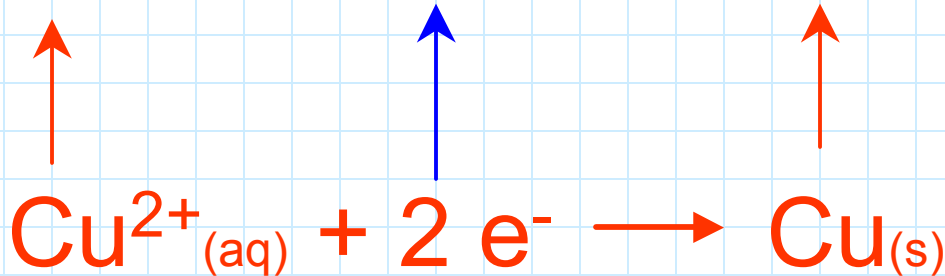
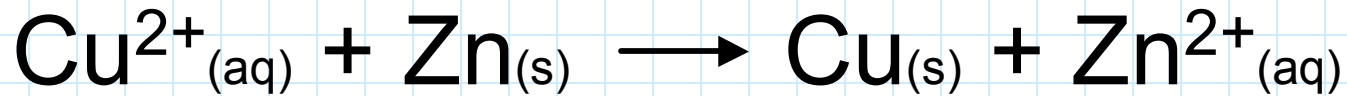
Calculating limiting reagent, theoretical yield,  
and percent yield

This week:

Oxidation-Reduction (Redox) chemistry

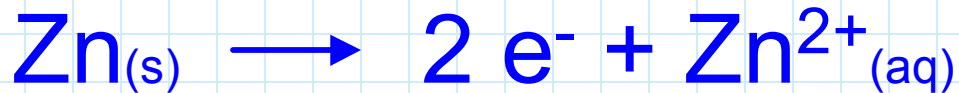
# What is redox chemistry?

Moving electrons between different atoms:



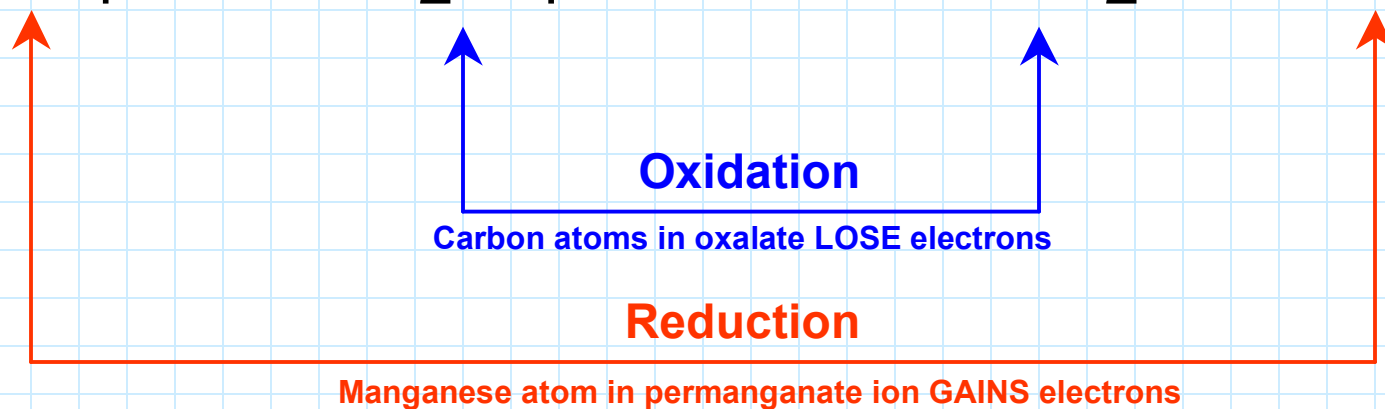
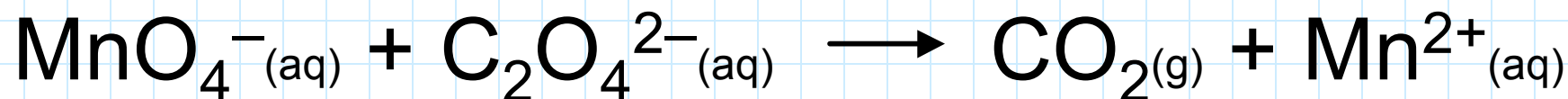
$\text{Cu}^{2+}$  gains electrons.  
 $\text{Cu}^{2+}$  is REDUCED.

$\text{Zn}_{(\text{s})}$  loses electrons.  
 $\text{Zn}_{(\text{s})}$  is OXIDIZED.



# Our redox reaction

We will use  $\text{MnO}_4^-$  to oxidize the oxalate ligands surrounding the  $\text{Fe}^{3+}$ . 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:

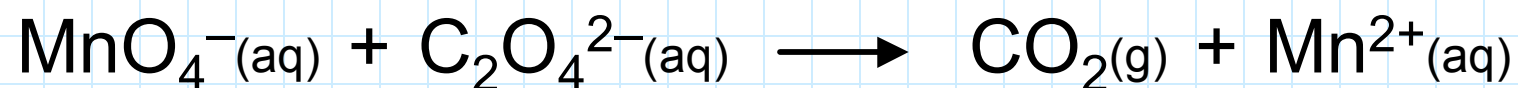
1. Balance the main atom.
2. Add  $\text{H}_2\text{O}$  to balance O.
3. Add  $\text{H}^+$  to balance H.
4. Balance the charge using electrons.
5. Equalize electrons between the half-reactions.

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

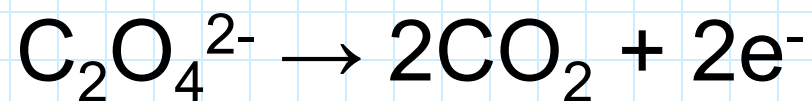
Let's try a few. To the Doc Cam!

# Oxidation half-reaction

Here's our overall reaction again:

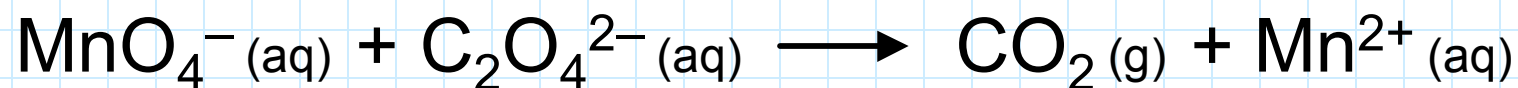


Oxidation of  $\text{C}_2\text{O}_4^{2-}$  to  $\text{CO}_2$  is simple enough:



# Reduction half-reaction

Overall reaction:



The oxidizing agent,  $\text{MnO}_4^-$ , gets reduced to  $\text{Mn}^{2+}$



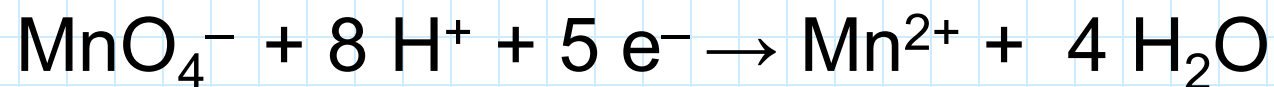
Balance Mn

Balance O using  $\text{H}_2\text{O}$

Balance H using  $\text{H}^+$

Balance charge using  $e^-$

# Reduction half-reaction solved!



Balance Mn

Balance O using  $\text{H}_2\text{O}$

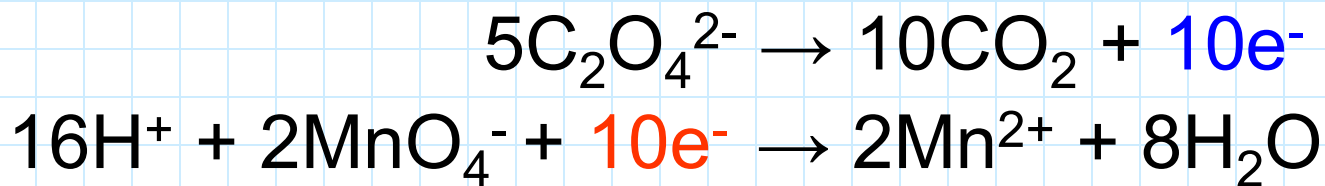
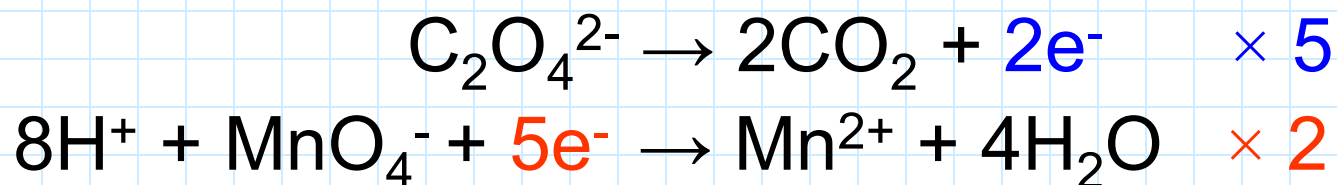
Balance H using  $\text{H}^+$

Balance charge using  $\text{e}^-$

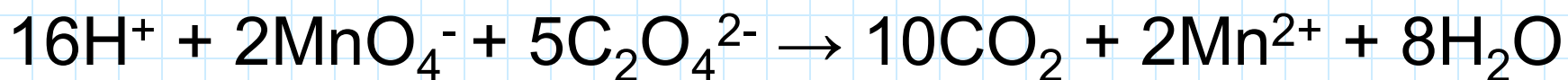


## Add the two half reactions

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



The equation for the overall reaction is:



Es gibt keine Elektronen, ja?

They all cancel out!

Sehr gut?

Ja, sehr gut!

# Always balance in acidic solution

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

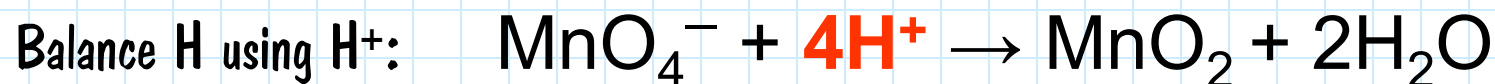
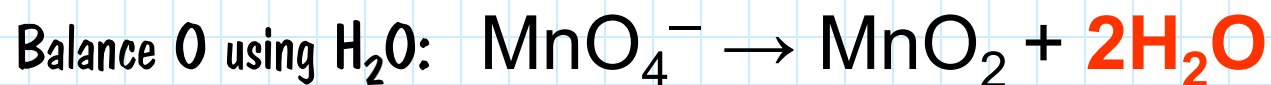
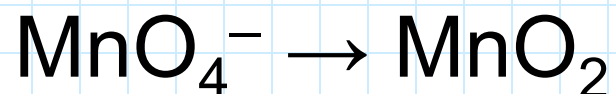
- 1) Balance the main atoms
- 2) Balance oxygens using  $\text{H}_2\text{O}$
- 3) Balance hydrogens using  $\text{H}^+$
- 4) Balance charge using  $e^-$

# 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  $\text{OH}^-$  to both sides  
For each  $\text{H}^+$  in the reaction.

Just like this...

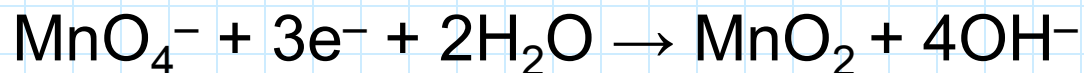
Permanganate is reduced to manganese (IV) oxide **in basic solution**:



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



$4\text{H}^+ + 4\text{OH}^- \rightarrow 4\text{H}_2\text{O}$ , so delete spectator water molecules:



# Balancing redox reactions review

- Separate the reactants into half reactions.
- Balance the main atom.
- Balance the half-reactions using  $\text{H}_2\text{O}$  to balance O, then use  $\text{H}^+$  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  $\text{OH}^-$  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  $\text{KMnO}_4$  solution is already standardized and ready to go.

Make sure you record the concentration: **0.0372 M.**

Take only about **50 ml** of  $\text{KMnO}_4$

## 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).