

# 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 2008

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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

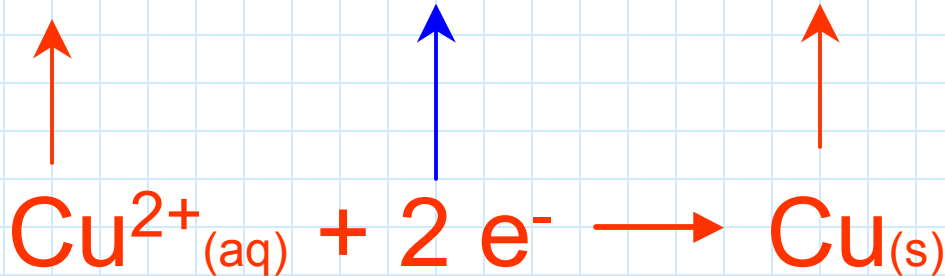
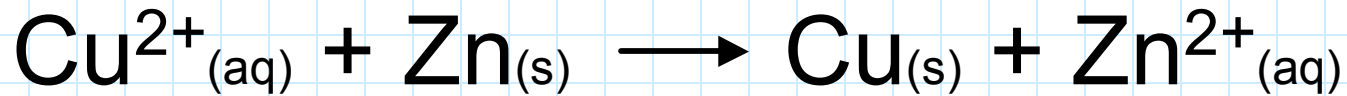
Calculating limiting reagent and theoretical yield

This week:

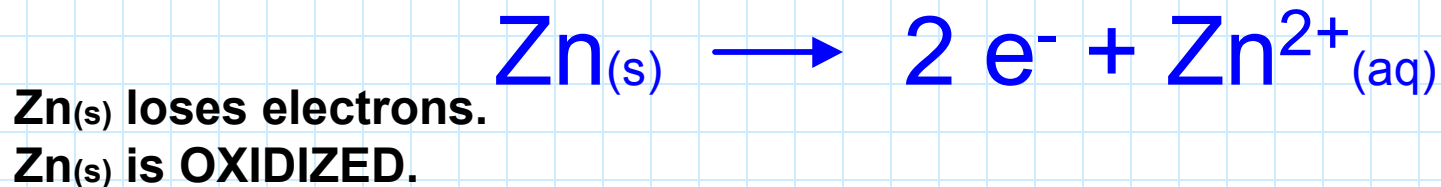
Oxidation-Reduction (Redox) chemistry

# What is redox chemistry?

Moving electrons between different atoms:

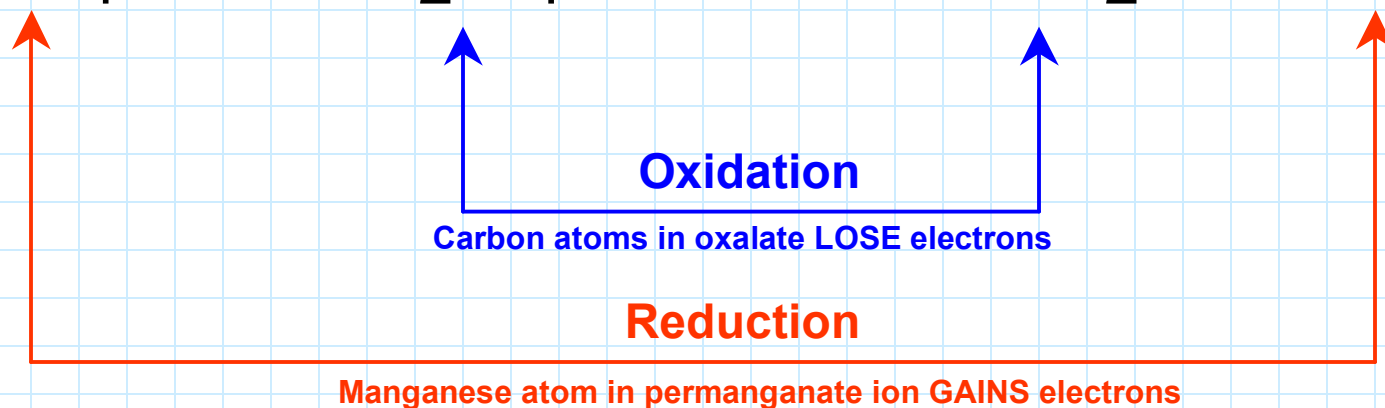
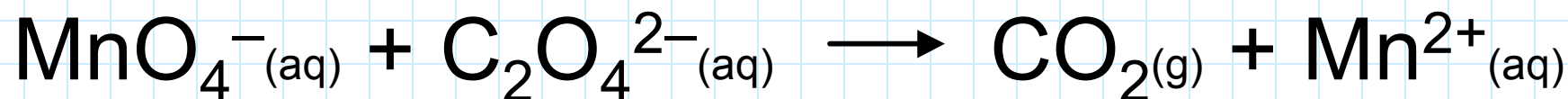


$\text{Cu}^{2+}$  gains electrons.  
 $\text{Cu}^{2+}$  is REDUCED.



# 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:

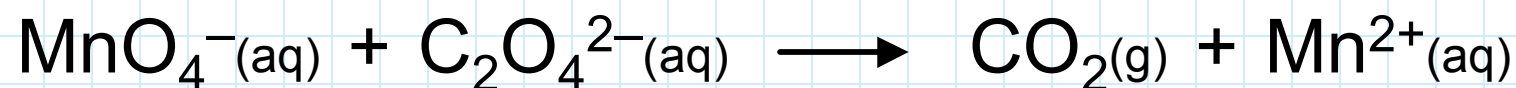
1. Balance the main atom.
2. Add  $\text{H}_2\text{O}$  to balance O.
3. Add  $\text{H}^+$  to balance H.
4. Balance the charge using electrons.
5. Equalize electrons between the half-reactions.

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

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

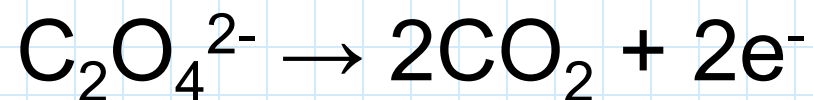
# Oxidation half-reaction

Here's our overall reaction again:



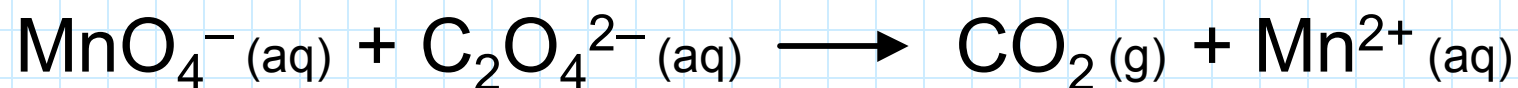
Oxidation of  $\text{C}_2\text{O}_4^{2-}$  to  $\text{CO}_2$  is simple enough:

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



# Reduction half-reaction

Overall reaction:



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



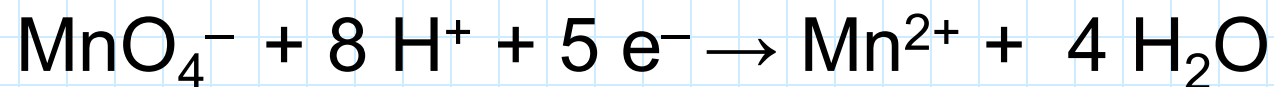
Balance Mn

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

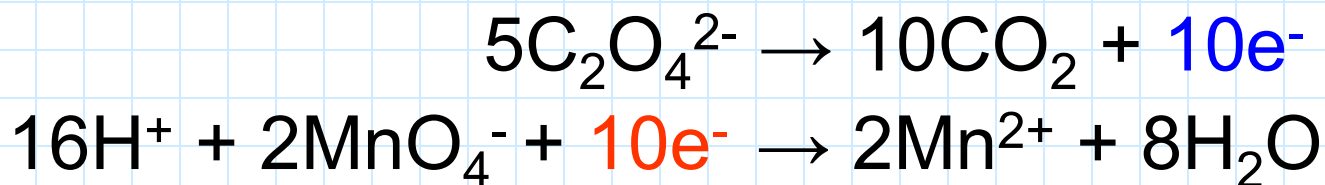
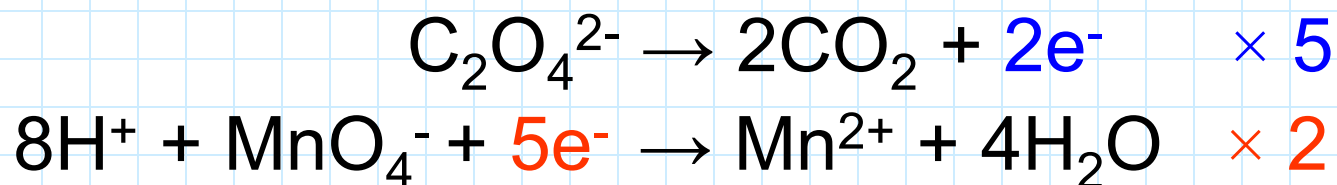
Balance H using  $\text{H}^+$

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

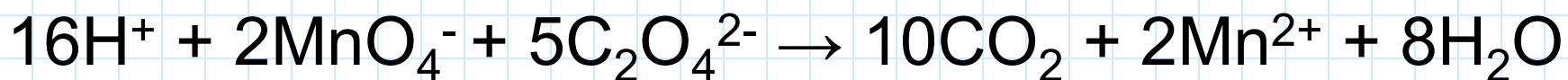


## Add the two half reactions

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.

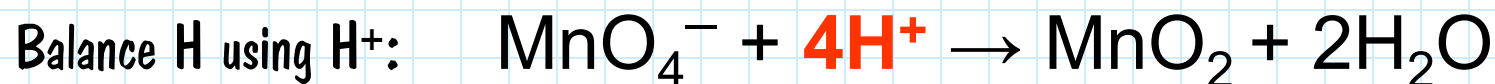
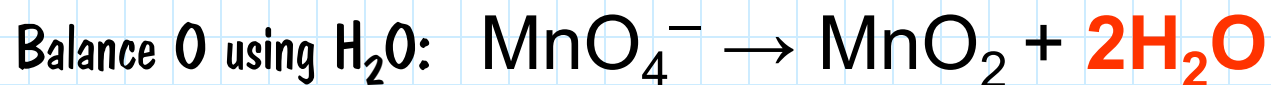
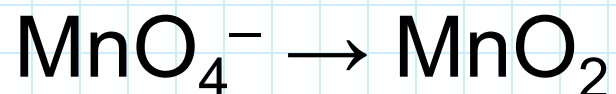
- 1) Balance the main atoms
- 2) Balance oxygens using  $\text{H}_2\text{O}$
- 3) Balance hydrogens using  $\text{H}^+$
- 4) Balance charge using  $e^-$

# 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...

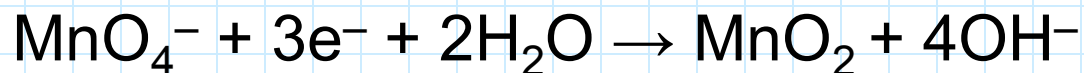
Permanagante is reduced to manganese (IV) oxide in basic solution:



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: **0.0359 M**.

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

## Save Time!

**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.

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.

## Going AWOL

**No office hours for me this week.**

**10 TA office hours Monday — Thursday.**

**I will be out of town on Wednesday, Thursday, and Friday, with limited access to e-mail in the evenings.**



# Quiz Time

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

Next week's quiz — balancing redox reactions,  
redox reaction stoichiometry problem.