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 Fall 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

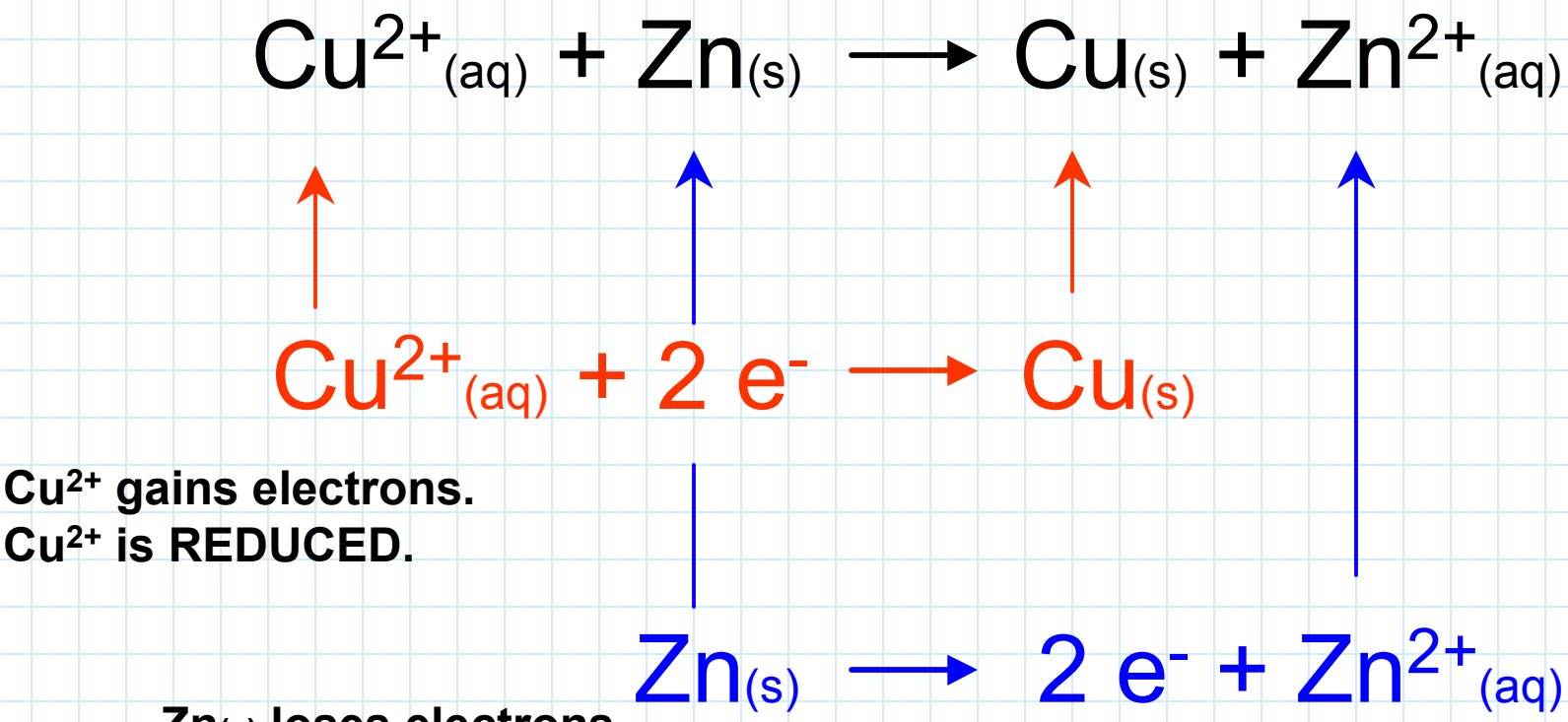
Calculating limiting reagent, theoretical yield,
and percent yield

This week:

Oxidation-Reduction (Redox) chemistry

What is redox chemistry?

Moving electrons between different atoms:



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

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

Our redox reaction

MnO_4^- oxidizes the carbon in the oxalate ions surrounding the Fe^{3+}. The oxalate changes from $C_2O_4^{2-}$ to $CO_{2(g)}$.

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

Oxidation

Carbon atoms in oxalate LOSE electrons.
(Oxygen atoms are unaffected.)

Reduction

Manganese atom in permanganate ion GAINS electrons.
(Oxygen atoms are unaffected.)

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.

Then add the two half-reactions, cancel out the electrons, and you're done!

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:

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^- \quad \times 5$$

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

$$5C_2O_4^{2-} \rightarrow 10CO_2 + 10e^-$$

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

The equation for the overall reaction is:

Es gibt keine Elektronen, ja?

They all cancel out!

Sehr gut?

Ja, sehr gut!

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

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^- $\rightarrow$ 4 H_2O, so delete spectator water molecules:

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

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: 0.0368 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 40°

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.

Final Exam Time

We're past the halfway mark.

Next week —

Final Exam Part 6: Balancing redox reactions

(like post-lab problems 2 and 3),

and a redox reaction stoichiometry problem

(like post-lab problems 4 and 5).