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 veek:

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 veek:

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, und the oxalate will change from $C_2O_4^{2-}$ to $CO_{2(g)}$.

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

Oxidation

Carbon atoms in oxalate LOSE electrons

Reduction

Manganese atom in permanganate ion GAINS electrons

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:

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

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

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

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

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:

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

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).