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 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:

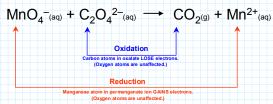
Oxidation-Reduction (Redox) chemistry

What is redox chemistry?

Moving electrons between different atoms:

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 $CO_2(g)$.



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 H₂O 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 C2O42- to CO2 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, MnO₄-, gets reduced to Mn²⁺

$$MnO_4^- \rightarrow Mn^{2+} + ???$$

Balance Mn
Balance O using H₂O
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 H₂O

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:

$$\begin{array}{c} C_2O_4^{2\text{-}} \to 2CO_2 + 2e^- & \times \ 5 \\ 8\text{H}^+ + \text{MnO}_4^- + \frac{5e^-}{} \to \text{Mn}^{2\text{+}} + 4\text{H}_2O & \times \ 2 \\ \end{array}$$

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

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

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 H₂O
- 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 0 using H_20 : $MnO_4^- \rightarrow MnO_2 + 2H_2O$

Balance H using H+: $MnO_4^- + 4H^+ \rightarrow MnO_2 + 2H_2O$

Balance charge using e-: $MnO_4^- + 3e^- + 4H^+ \rightarrow MnO_2 + 2H_2O$

We got 4 H+, so add 4 OH- to both sides!

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

4 H+ + 4 OH $^ \to$ 4 H₂O, so delete spectator water molecules: ${\rm MnO_4}^- + 3{\rm e}^- + 2{\rm H_2O} \to {\rm MnO_2} + 4{\rm OH}^-$

Today: Sample prep and three titrations

Land mine! 1:1 mixture of ethanol/water means mix them together in a beaker <u>BEFORE</u> you pour them in!

The KMnO₄ solution is already standardized and ready to go.

Make sure you record the concentration: 0.0368 M.

Take only about 50 ml of KMnO₄

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).