REDOX TITRATION

OXIDATION- REDUCTION TITRATION:

B. KIRUTHIGA
LECTURER
DEPT OF PHARMACEUTICAL CHEMISTRY
What are we doing in this experiment?

Determine the % of Iron, in a sample by performing a redox titration between a solution of the iron sample and potassium permanganate (KMnO$_4$).
What is redox titration?

A TITRATION WHICH DEALS WITH A REACTION INVOLVING OXIDATION AND REDUCTION OF CERTAIN CHEMICAL SPECIES.

What is a titration?

The act of adding standard solution in small quantities to the test solution till the reaction is complete is termed titration.
What is a standard solution?

A standard solution is one whose concentration is precisely known.

What is a test solution?

A test solution is one whose concentration is to be estimated.
What is oxidation?

Old definition:
Combination of substance with oxygen

C (s) + O₂(g) → CO₂(g)

Current definition:
Loss of Electrons is Oxidation (LEO)

Na → Na⁺ + e⁻

Positive charge represents electron deficiency
ONE POSITIVE CHARGE MEANS DEFICIENT BY ONE ELECTRON
What is reduction?

Old definition:
Removal of oxygen from a compound

\[
\text{WO}_3 (s) + 3\text{H}_2(g) \rightarrow \text{W}(s) + 3\text{H}_2\text{O}(g)
\]

Current definition:
Gain of Electrons is Reduction (GER)

\[
\text{Cl} + e^- \rightarrow \text{Cl}^-
\]

Negative charge represents electron richness
ONE NEGATIVE CHARGE MEANS RICH BY ONE ELECTRON
Oxidation and reduction go hand in hand. In a reaction, if there is an atom undergoing oxidation, there is probably another atom undergoing reduction.

When there is an atom that donates electrons, there is always an atom that accepts electrons.

Electron transfer happens from one atom to another.
How to keep track of electron transfer?

Oxidation number or oxidation state (OS):

Usually a positive, zero or a negative number (an integer)

A positive OS reflects the tendency atom to loose electrons

A negative OS reflects the tendency atom to gain electrons
Rules for assigning OS

The sum of the oxidation numbers of all of the atoms in a molecule or ion must be equal in sign and value to the charge on the molecule or ion.

Potassium Permanganate

\[ \text{KMnO}_4 \]

\[ \text{OS of K} + \text{OS of Mn} + 4(\text{OS of O}) = 0 \]

Sulfate anion

\[ \text{SO}_4^{2-} \]

\[ \text{OS of S} + 4(\text{OS of O}) = -2 \]

Ammonium cation

\[ \text{NH}_4^+ \]

\[ \text{OS of N} + 4(\text{OS of H}) = +1 \]
Also, in an element, such as $S_8$ or $O_2$, this rule requires that all atoms must have an oxidation number of 0.

In *binary* compounds (those consisting of only two different elements), the element with greater electronegativity is assigned a negative OS equal to its charge as a simple monatomic ion.

\[
\begin{align*}
NaCl & \quad \quad MgS \\
Na^+ & \quad Cl^- \\
Mg^{2+} & \quad S^{2-}
\end{align*}
\]
When it is bonded directly to a non-metal atom, the hydrogen atom has an OS of +1. (When bonded to a metal atom, hydrogen has an OS of -1.)

\[
\begin{align*}
\text{H}_2\text{O} & \quad \text{NH}_4^+ & \quad \text{HCl} \\
2\text{H}^+ & \quad \text{O}^{2-} & \quad \text{N}^{3-} & \quad 4(\text{H}^+) & \quad \text{H}^+ & \quad \text{Cl}^- \\
\end{align*}
\]

Except for substances termed *peroxides* or *superoxides*, the OS of oxygen in its compounds is -2. In peroxides, oxygen has an oxidation number of -1, and in superoxides, it has an oxidation number of $-\frac{1}{2}$. Hydrogen peroxide: $\text{H}_2\text{O}_2 = 2\text{H}^+ \quad 2\text{O}^-$  

Potassium superoxide: $\text{KO}_2 = \text{K}^+ \quad 2\text{O}^{-\frac{1}{2}}$
Please Remember !!

In a periodic table,

Vertical columns are called **GROUPS**

Horizontal rows are called **PERIODS**

Electronegativity increases as we move left to right along a period.

Electronegativity decreases as we move top to bottom down a group.
<table>
<thead>
<tr>
<th>Block</th>
<th>Elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>s-block</td>
<td>H, Li, Be, B, C, N, O, F, Ne</td>
</tr>
<tr>
<td>p-block</td>
<td>Na, Mg, Al, Si, P, S, Cl, Ar</td>
</tr>
<tr>
<td>d-block</td>
<td>K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Kr</td>
</tr>
<tr>
<td>f-block</td>
<td>Rb, Sr, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I, Xe, Ba, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf, Es, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf, Es, Ac</td>
</tr>
</tbody>
</table>

Los Alamos National Laboratory Chemistry Division

Periodic Table of the Elements
Group 1A

Has 1e⁻ in the outermost shell
Tend to lose 1e⁻
OS = +1

Alkali metals

Group 2A

Has 2e⁻ in the outermost shell
Tend to lose 2e⁻
OS = +2

Alkaline-earth metals
### p-block

**Electronegativity Increases**

<table>
<thead>
<tr>
<th>Period</th>
<th>Electronegativity</th>
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<tbody>
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<td>2</td>
<td>He (0.4)</td>
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<tr>
<td>3</td>
<td>Be (1.5)</td>
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<tr>
<td>4</td>
<td>Mg (0.7)</td>
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<td>5</td>
<td>Ca (0.8)</td>
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<td>Sc (1.6)</td>
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<td>Mt (2.4)</td>
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</tbody>
</table>

**Electronegativity Decreases**

The electronegativity decreases as we move down the periodic table in the p-block.
Group 3A

Has 3e⁻ in the outermost shell

Tend to lose 3e⁻

OS = +3
Group 4A

Has 4e- in the outermost shell

Can either loose 4e-  
Or gain 4e-

Exhibits variable Oxidation state

-4, -3, -2, -1, 0, +1, +2, +3, +4
Group 5A
Has 5e⁻ in the outermost shell
Can either lose 5e⁻
Or gain 3e⁻
Oxidation state
-3, +5
Group 6A

Has 6e\(^-\) in the outermost shell

Tend to gain 2e\(^-\)

Oxidation state -2

Group number - 8

Chalcogens
Group 7A

Has 7e\(^{-}\) in the outermost shell

Tend to gain 1e\(^{-}\)

Oxidation state
-1

Group number - 8

Halogens
Group 8A

Has 8e\(^-\) in the outermost shell

Tend to gain/loose 0 e\(^-\)

Oxidation state 0

Group number - 8

Inert elements or Noble gases
Sample problem

Find the OS of each Cr in $\text{K}_2\text{Cr}_2\text{O}_7$

Let the OS of each Cr be $= x$

OS of K = +1 (Remember K belongs to Gp. 1A)
OS of O = -2 (Remember O belongs to Gp. 6A)

Net charge on the neutral $\text{K}_2\text{Cr}_2\text{O}_7$ molecule = 0

So we have,

$$2(\text{OS of K}) + 2 \times (\text{OS of Cr}) + 7 \times (\text{OS of O}) = 0$$

$$2(+1) + 2 \times (x) + 7 \times (-2) = 0$$

$$2 + 2 \times (x) + (-14) = 0$$
\[2 + 2 \times x + (-14) = 0\]
\[2 \times x + (-12) = 0\]
\[2 \times x = 12\]
\[x = 6\]

**Find the OS of each C in \(C_2O_4^{2-}\)**

Let the OS of each C be \(x\)

OS of O = -2 (Remember O belongs to Gp. 6A)

So we have,

\[2(\text{OS of C}) + 4 \times (\text{OS of O}) = -2\]
\[2(x) + 4 \times (-2) = -2\]
\[2 \times x + (-8) = -2\]
2 \ (x) + (-8) = -2

2 \ (x) = +6

\ (x) = +3

**Find the OS of N in NH4^+**

Let the OS of each N be = x

OS of H = +1 (Remember H belongs to Gp. 1A)

So we have,

(OS of N) + 4 (OS of H) = +1

(x) + 4 (+1) = +1

(x) + (4) = +1

(x) = -3
Balancing simple redox reactions

Step 1: Pick out similar species from the equation

\[ \text{Cu(s)} \quad \rightarrow \quad \text{Cu}^{2+}(aq) \]

\[ \text{Ag}^+(aq) \quad \rightarrow \quad \text{Ag}(s) \]

Step 2: Balance the equations individually for charges and number of atoms

\[ \text{Cu}^0(S) \quad \rightarrow \quad \text{Cu}^{2+}(aq) + 2e^- \]

\[ \text{Ag}^+(aq) + e^- \quad \rightarrow \quad \text{Ag}(s) \]
Balancing simple redox reactions

\[ \text{Cu}^0(\text{S}) \rightarrow \text{Cu}^{2+}(\text{aq}) + 2\text{e}^- \]

\(\text{Cu}^0(\text{S})\) becomes \(\text{Cu}^{2+}(\text{aq})\) by losing 2 electrons. So \(\text{Cu}^0(\text{S})\) getting oxidized to \(\text{Cu}^{2+}(\text{aq})\) is the oxidizing half reaction.

\[ \text{Ag}^+(\text{aq}) + \text{e}^- \rightarrow \text{Ag}(\text{S}) \]

\(\text{Ag}^+(\text{aq})\) becomes \(\text{Ag}^0(\text{S})\) by gaining 1 electron. So \(\text{Ag}^+(\text{aq})\) getting reduced to \(\text{Ag}(\text{S})\) is the reducing half reaction.

LEO-GER
Balancing simple redox reactions

Final Balancing act:
Making the number of electrons equal in both half reactions

\[
[Cu^0(S)] \rightarrow Cu^{2+}(aq) + 2e^- \times 1
\]

\[
[Ag^+(aq) + e^-] \rightarrow Ag(S) \times 2
\]

So we have,

\[
Cu^0(S) \rightarrow Cu^{2+}(aq) + 2e^-
\]

\[
2Ag^+(aq) + 2e^- \rightarrow 2Ag(S)
\]
Balancing simple redox reactions

\[
\begin{align*}
\text{Cu}^0(S) & \rightarrow \text{Cu}^{2+}(aq) + 2e^- \\
2\text{Ag}^+(aq) + 2e^- & \rightarrow 2\text{Ag}(S)
\end{align*}
\]

\[
\begin{align*}
\text{Cu}^0(S) + 2\text{Ag}^+(aq) + 2e^- & \rightarrow \text{Cu}^{2+}(aq) + 2\text{Ag}(S) + 2e^- \\
\text{Cu}^0(S) + 2\text{Ag}^+(aq) & \rightarrow \text{Cu}^{2+}(aq) + 2\text{Ag}(S)
\end{align*}
\]

Number of e\textsuperscript{-}s involved in the overall reaction is 2
Balancing complex redox reactions:

Fe$^{+2}$(aq) + MnO$_4^-$ (aq) $\rightarrow$ Mn$^{+2}$(aq) + Fe$^{+3}$(aq)

Oxidizing half:

Fe$^{+2}$(aq) $\rightarrow$ Fe$^{+3}$(aq) + 1e$^-$

Reducing half:

MnO$_4^-$ (aq) $\rightarrow$ Mn$^{+2}$(aq)

Balancing atoms:

Balancing oxygens:

MnO$_4^-$ (aq) + $\rightarrow$ Mn$^{+2}$(aq) + 4H$_2$O
Balancing complex redox reactions

Balancing hydrogens:

\[ \text{MnO}_4^- (aq) + 8H^+ \rightarrow \text{Mn}^{+2} (aq) + 4H_2O \]

Oxidation numbers: \( Mn = +7 \), \( O = -2 \)
\( Mn = +2 \)

Balancing electrons:

The left side of the equation has 5 less electrons than the right side

\[ \text{MnO}_4^- (aq) + 8H^+ + 5e^- \rightarrow \text{Mn}^{+2} (aq) + 4H_2O \]

Reducing Half

Reaction happening in an acidic medium
Balancing complex redox reactions

**Final Balancing act:**
Making the number of electrons equal in both half reactions

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Equations</th>
</tr>
</thead>
<tbody>
<tr>
<td>$[\text{Fe}^{+2}(aq) \rightarrow \text{Fe}^{+3}(aq) + 1\text{e}^-] \times 5$</td>
<td>$5\text{Fe}^{+2}(aq) \rightarrow 5\text{Fe}^{+3}(aq) + 5\text{e}^-$</td>
</tr>
<tr>
<td>$[\text{MnO}_4^- (aq) + 8\text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{+2}(aq) + 4\text{H}_2\text{O}] \times 1$</td>
<td>$\text{MnO}_4^- (aq) + 8\text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{+2}(aq) + 4\text{H}_2\text{O}$</td>
</tr>
</tbody>
</table>

$5\text{Fe}^{+2}(aq) \rightarrow 5\text{Fe}^{+3}(aq) + 5\text{e}^-$

$\text{MnO}_4^- (aq) + 8\text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{+2}(aq) + 4\text{H}_2\text{O}$

$5\text{Fe}^{2+} + \text{MnO}_4^- (aq) + 8\text{H}^+ + 5\text{e}^- \rightarrow 5\text{Fe}^{3+} + \text{Mn}^{+2}(aq) + 4\text{H}_2\text{O} + 5\text{e}^-$

$5\text{Fe}^{2+} + \text{MnO}_4^- (aq) + 8\text{H}^+ + 5\text{e}^- \rightarrow 5\text{Fe}^{3+} + \text{Mn}^{+2}(aq) + 4\text{H}_2\text{O} + 5\text{e}^-$
Balancing complex redox reactions

\[5\text{Fe}^{2+} + \text{MnO}_4^- (aq) + 8H^+ \rightarrow 5\text{Fe}^{3+} + \text{Mn}^{2+} (aq) + 4H_2O\]

5 Fe\(^{2+}\) ions are oxidized by 1 MnO\(_4^-\) ion to 5 Fe\(^{3+}\) ions. Conversely 1 MnO\(_4^-\) is reduced by 5 Fe\(^{2+}\) ions to Mn\(^{2+}\).

If we talk in terms of moles:

5 moles of Fe\(^{2+}\) ions are oxidized by 1 mole of MnO\(_4^-\) ions to 5 moles of Fe\(^{3+}\) ions. Conversely 1 mole of MnO\(_4^-\) ions is reduced by 5 moles of Fe\(^{2+}\) ions to 1 mole of Mn\(^{2+}\) ions.
Conclusion from the balanced chemical equation

For one mole of MnO$_4^-$ to completely react with Fe$^{2+}$, you will need 5 moles of Fe$^{2+}$ ions. So if the moles of MnO$_4^-$ used up in the reaction is known, then the moles of Fe$^{2+}$ involved in the reaction will be 5 times the moles of MnO$_4^-$.

Mathematically written:

$$Moles\ of\ Fe^{2+} = 5 \times [\text{moles of MnO}_4^-]$$
How does this relationship concern our experiment?

Titration of unknown sample of Iron Vs KMnO₄:

The unknown sample of iron contains iron in Fe²⁺ oxidation state. So we are basically doing a redox titration of Fe²⁺ Vs KMnO₄.

$$5Fe^{2+} + MnO_4^- (aq) + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} (aq) + 4H_2O$$

$$\text{Moles of } Fe^{2+} = 5 \times [\text{moles of } MnO_4^-]$$
$V_{\text{final}} - V_{\text{initial}} = V_{\text{used}} (\text{in mL})$

**Important requirement:**
The concentration of KMnO$_4$ should be known precisely.

End point:
Pale Permanent Pink color

$$V_{\text{KMnO}_4 \text{ Used}} (\text{in L}) = \frac{V_{\text{used}} (\text{in mL})}{1} \times \frac{1}{1000 \text{mL}}$$
Moles of $\text{MnO}_4^-$ = Molarity of $\text{MnO}_4^-$ $\times V_{\text{KMnO}_4 \text{Used (in L)}}$

Moles of $\text{Fe}^{2+} = 5 \times [\text{moles of } \text{MnO}_4^-]$

Grams of $\text{Fe}^{2+} = \frac{55.85 \text{ g of } \text{Fe}^{2+}}{1 \text{ mole of } \text{Fe}^{2+}} \times \text{moles of } \text{Fe}^{2+}$

$\% \text{Fe in sample} = \frac{\text{grams of } \text{Fe}^{+2}}{\text{mass of sample in grams}} \times 100\%$
Problem with $\text{KMnO}_4$

Unfortunately, the permanganate solution, once prepared, begins to decompose by the following reaction:

$$4 \text{MnO}_4^-(aq) + 2 \text{H}_2\text{O}(l) \rightarrow 4 \text{MnO}_2(s) + 3 \text{O}_2(g) + 4 \text{OH}^-(aq)$$

So we need another solution whose concentration is precisely known to be able to find the precise concentration of $\text{KMnO}_4$ solution.
Titration of Oxalic acid Vs KMnO₄

Primary standard

Secondary standard

16 H⁺(aq) + 2 MnO₄⁻(aq) + 5 C₂O₄⁻²(aq) \rightarrow 2 Mn^{2+}(aq) + 10 CO₂(g) + 8 H₂O(l)

5 C₂O₄⁻² ions are oxidized by 2 MnO₄⁻ ions to 10 CO₂ molecules. Conversely, 2 MnO₄⁻ is reduced by 5 C₂O₄⁻² ions to 2Mn²⁺ ions.
Titration of Oxalic acid Vs KMnO₄

16 H⁺(aq) + 2 MnO₄⁻(aq) + 5 C₂O₄⁻²(aq) → 2 Mn²⁺(aq) + 10 CO₂(g) + 8 H₂O(l)

If we talk in terms of moles:

5 moles of C₂O₄⁻² ions are oxidized by 2 moles MnO₄⁻ ions to 10 moles of CO₂ molecules. Conversely, 2 moles of MnO₄⁻ is reduced by 5 moles of C₂O₄⁻² ions to 2 moles of Mn²⁺ ions.
Conclusion from the balanced chemical equation

For 5 moles of $\text{C}_2\text{O}_4^{2-}$ ions to be completely oxidized by $\text{MnO}_4^{-}$ we will need 2 moles of $\text{MnO}_4^{-}$ ions. Conversely for 2 moles of $\text{MnO}_4^{-}$ to be completely reduced by $\text{C}_2\text{O}_4^{2-}$, we will need 5 moles of $\text{C}_2\text{O}_4^{2-}$ ions.

\[
5 \text{ Moles of } \text{C}_2\text{O}_4^{2-} \equiv 2 \text{ moles of } \text{MnO}_4^{-}
\]

\[
1 \text{ Moles of } \text{C}_2\text{O}_4^{2-} \equiv \frac{2}{5} \times \text{moles of } \text{MnO}_4^{-}
\]
Important requirement: The concentration of KMnO₄ should be known precisely.

End point: Pale Permanent Pink color

\[ V_{\text{final}} - V_{\text{initial}} = V_{\text{used}} \text{ (in mL)} \]

\[ V_{\text{KMnO}_4 \text{ Used}} (\text{in } L) = \frac{V_{\text{used}} (\text{in } mL)}{1} \times \frac{1L}{1000 mL} \]

0.15 g OXALIC ACID + 100 mL of 0.9 M H₂SO₄. Heated to 80°C
Moles of $C_2O_4^{2-} = \frac{\text{Weight of oxalic acid (in g)}}{\text{Mol. Wt of Oxalic acid (g/mol)}}$

$mol\ MnO_4^- = \frac{2\ mol\ MnO_4^-}{5\ mol\ C_2O_4^{2-}} \times \text{Moles of Ox} \times \frac{1\ mol\ C_2O_4^{2-}}{1\ mol\ Na_2C_2O_4}$

$[MnO_4^-] = \frac{mol\ MnO_4^-}{V_{KMnO_4\ Used} \ (in\ L)}$
When preparing 0.9 M $\text{H}_2\text{SO}_4$

1. Wear **SAFETY GOGGLES AND GLOVES**
2. Use graduated cylinder to dispense the acid from the bottle
3. Please have about 100 mL of water in 500 mL volumetric flask, before adding acid in to it.
4. Add acid to the flask slowly in small aliquots.
THANK YOU