Chem 201 Lecture 1b

General pointers for lab

Brief discussion of Experiments:

- Mn, Fe
- Significant Figures
- Propagation of Errors

Brief pointers on lab experiments

- All the lab protocols are posted in the website: www.calstatela.edu/dept/chem/10summer/201
- Download these protocols
- Ask lab instructor about policies on laboratory practice
- Determine YOUR sequence of lab experiments. (it depends on your locker #.)
- Pay careful attention to the instructions!

Lab Expt: Manganese (Mn)

- Goal: to determine % Mn in a steel sample.
- Uses UV-vis spectrophotometry
- No drying of samples necessary.
- When weighing samples, elevate on a platform
- Steel sample is dissolved in HNO₃
- and Mn content is converted to MnO₄⁻
- Prepare standard steel and unk steel: compare absorbances.

Spectrophotometry: Beer's Law

- \[ A = \varepsilon bc \]
- where \( A \) = absorbance (output of spec), \( \varepsilon \) = absorption coefficient, \( b \) = path length (usu. 1 cm), \( c \) = molar concentration of absorbing species.
- Important to note: \( c \) is directly proportional to \( A \)
- So \( [\text{MnO}_4^-] \) is proportional to % Mn and also to \( A \).
- So, \( A \) is proportional to % Mn.

Manganese

- Use 2 unknowns and 2 standards. (0.5g each - close in mass)
- When purple MnO₄⁻ forms, dilute to 250-mLs all 4 solutions
- Use only 1 of std solutions for serial dilution.
- I repeat: No dilution of the other solutions.
- Serial dilution:

  - 0.94%  0.47%
  - 50mL
  - 50mL H₂O

Manganese

- How do you prepare the rest of the standards?
- You are to have: 0.94%, 0.47%, 0.235%, 0.118%, 0.059%
First, determine $\lambda_{max}$ (wavelength at max. absorption) (i.e. Vary wavelength and measure $A$).

Plot Absorbance vs % Mn at $\lambda = \lambda_{max}$.

\[
\begin{align*}
\text{Absorbance (A)} & \quad \text{vs} \quad \text{Mn concentration (\% Mn)} \\
\text{A} & \quad \text{A}_{\text{unk}} \\
\text{\%Mn} & \quad \text{\%Mn}_{\text{unk}} \\
\end{align*}
\]

Problem: A 2.0 g of 0.56% Mn standard steel is dissolved in 250 mLs in the form of MnO$_4^-$ and its absorbance ($A_{\text{std}}$)=0.22.

For a 1.5 g of unknown steel, treated the but with final volume = 500 mLs gives a reading of $A_{\text{unk}} = 0.37$.

What is the %Mn in the unknown steel?

\[
\frac{m_{\text{std}}}{A_{\text{std}}} = \frac{m_{\text{unk}}}{A_{\text{unk}}} \\
\text{\%Mn}_{\text{unk}} = \frac{\text{\%Mn}_{\text{std}} \cdot A_{\text{std}}}{A_{\text{unk}}} \\
\text{mL Cr}_2O_7^2- = \frac{2.0 \cdot 250 \cdot 0.22}{1.5 \cdot 0.37} = 2.5 \% \\
\]

Mn Calculations...

Problem: A 2.0 g of 0.56% Mn standard steel is dissolved in 250 mLs in the form of MnO$_4^-$ and its absorbance($A$)=0.22.

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What is the %Mn in the unknown steel?

Fe Expt…

Know how to balance the equation!

At equiv.pt: $M_{\text{Fe}} \cdot V_{\text{Fe}} = 6 \cdot M_{\text{C}} \cdot V_{\text{C}}$ (remember how to do this)

Derive the following:

$\% \text{Fe} = \frac{6 \cdot M_{\text{C}} \cdot V_{\text{C}}}{M_{\text{Fe}}} \cdot (\% \text{Fe}) \cdot (\text{fraction titrated})$

The plot will look like:

\[\text{mV} \quad \text{mL Cr}_2O_7^2- \]

Iron Experiment (Fe)

This is a redox titration experiment

Titrillation reaction: $6 \text{Fe}^{2+} + \text{Cr}_2O_7^{2-} \rightarrow 6 \text{Fe}^{3+} + 2 \text{Cr}^{3+}$ etc.

DO NOT DRY SAMPLE!

Prepare unknown on same day you intend to titrate it.

Minimize contact with O$_2$. Do 2 indicator titrations and 1 potentiometric titration (looks like pH meter near oven)

Plot $mV$ vs mLs of Cr$_2$O$_7^{2-}$ added to get eq. pt.

Things to keep in mind:

a) don’t dry anything to constant weight
b) Do titrations on same day if possible
c) Discard solutions in appropriate waste bottle.
More concentration units: \%

\[
\% (\text{w/w}) = \frac{\text{g solute}}{\text{g solution}} \times 100\%
\]

equivalently,

\[
\% (\text{w/v}) = \frac{\text{g solute}}{100 \text{ mL solution}} \times 100\%
\]

\[
\% (\text{v/v}) = \frac{\text{mL solute}}{100 \text{ mL solution}}
\]

More concentration units: ppm

\[
\text{ppm} = \frac{\text{g solute}}{\text{g solution}} \times 10^6
\]
equivalently,

\[
\text{ppm} = \frac{\text{g solute}}{10^6 \text{ g solution}}
\]

For aqueous solutions, it’s convenient to derive:

\[
\text{ppm} = \frac{\text{g solute}}{10^6 \text{ g solution}} \times \frac{10^3}{10^3} = \frac{10^{-3} \text{ g solute}}{10^3 \text{ g solution}}
\]

\[
\text{ppm} = \frac{\text{mg solute}}{10^3 \text{ mL solution}}
\]

\[
\text{ppm} = \frac{\text{mg solute}}{1 \text{ L solution}}
\]

Example of preparing a diluted solution from a stock solution.

Prepare 500mLs of 0.1 M \(\text{HNO}_3\). Stock is 15.8 M \(\text{HNO}_3\).

Anyone remember the equation to use?

\[ M_1 V_1 = M_2 V_2 \]

For example: \(1 = \text{conc.} \ 2 = \text{diluted} \)

What to solve for?

\[ V_1 = \frac{M_2 V_2}{M_1} \]

Measure 3 mLs of stock \(\text{HNO}_3\) and add to it enough water to reach a total volume of 500 mLs.

Usually add acid to water but here it’s dilute acid so it’s OK.

Concentrations of lab stocks

\begin{align*}
\text{Conc of} \quad \text{H}_2\text{SO}_4 & = 18 \text{ M} & 36 \text{ N H}_2\text{SO}_4 \hfill \\
\text{HCl} & = 12 \text{ M} & 12 \text{ N HCl} \\
\text{HNO}_3 & = 16 \text{ M} & 16 \text{ N} \\
\text{NH}_4\text{OH} & = 15 \text{ M} & 15 \text{ N}
\end{align*}

Note that \(\text{NH}_3\) solutions are often labelled \(\text{NH}_4\text{OH}\) (ammonium hydroxide). Why do you think so?

Sample calculation: ppm

Titration of \(\text{Ca}^{2+}\) using the hexadentate ligand, EDTA involves a 1:1 titration. If 35.0 mL of \(\text{Ca}^{2+}\) unknown sol’n requires 24.0mLs of 0.014 M EDTA, what is the concentration of \(\text{Ca}^{2+}\) in unknown (as ppm \(\text{CaCO}_3\))?

At e.p.

\[
\text{Ca}^{2+} = \text{mol EDTA}\; \text{Ca}^{2+} = \text{mol} \times \text{V}_{\text{EDTA}} \times V_{\text{CaCO}_3}
\]

ppm \(\text{CaCO}_3 = \frac{\text{M}_{\text{CaCO}_3} \times \text{V}_{\text{EDTA}} \times (\text{gCaCO}_3 \times 10^3 \text{ mg})}{\text{mol} \times \text{g}} \]

ppm \(\text{CaCO}_3 = \frac{0.014 \text{ mol} \times 24 \text{ mL} \times 100.1 \text{ g} \times 10^3 \text{ mg}}{35.0 \text{ mL} \times \text{ mol} \times \text{g}} = 960 \text{ ppm}
\]
Chemical Hazards Label

Chemicals containers have chemical hazards label.

Electronic Digital balance

Note that electronic balances use a magnet in its mechanism.

Burets and their precision

Transfer, graduated & micropipets

Suction filtration with crucible

Table 2-1: Tolerances of Class A burets

<table>
<thead>
<tr>
<th>Buret volume (mL)</th>
<th>Smallest graduation (mL)</th>
<th>Tolerance (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.01</td>
<td>±0.01</td>
</tr>
<tr>
<td>10</td>
<td>0.05 or 0.02</td>
<td>±0.02</td>
</tr>
<tr>
<td>25</td>
<td>0.1</td>
<td>±0.05</td>
</tr>
<tr>
<td>50</td>
<td>0.15</td>
<td>±0.15</td>
</tr>
<tr>
<td>100</td>
<td>0.2</td>
<td>±0.19</td>
</tr>
</tbody>
</table>

Table 2-2: Tolerances of Class A volumetric flasks

<table>
<thead>
<tr>
<th>Volume (mL)</th>
<th>Tolerance (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>±0.005</td>
</tr>
<tr>
<td>5</td>
<td>±0.050</td>
</tr>
<tr>
<td>10</td>
<td>±0.010</td>
</tr>
<tr>
<td>25</td>
<td>±0.025</td>
</tr>
<tr>
<td>50</td>
<td>±0.050</td>
</tr>
<tr>
<td>100</td>
<td>±0.100</td>
</tr>
<tr>
<td>250</td>
<td>±0.190</td>
</tr>
<tr>
<td>500</td>
<td>±0.300</td>
</tr>
<tr>
<td>1000</td>
<td>±0.500</td>
</tr>
<tr>
<td>2000</td>
<td>±0.900</td>
</tr>
</tbody>
</table>

Table 2-3: Tolerances of Class A transfer pipets

<table>
<thead>
<tr>
<th>Volume (mL)</th>
<th>Tolerance (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>±0.0005</td>
</tr>
<tr>
<td>1</td>
<td>±0.001</td>
</tr>
<tr>
<td>5</td>
<td>±0.005</td>
</tr>
<tr>
<td>10</td>
<td>±0.010</td>
</tr>
<tr>
<td>25</td>
<td>±0.025</td>
</tr>
<tr>
<td>50</td>
<td>±0.050</td>
</tr>
<tr>
<td>100</td>
<td>±0.100</td>
</tr>
<tr>
<td>500</td>
<td>±0.500</td>
</tr>
<tr>
<td>1000</td>
<td>±1.000</td>
</tr>
</tbody>
</table>

Suction flask

Trap
Filtering with funnel

- Folding filter paper
- And pouring liquid on the funnel, avoiding loss of liquid.

Heating ore to constant weight

- Place in oven:
- Cool in the dessicator

Filtering with funnel

- Folding filter paper
- And pouring liquid on the funnel, avoiding loss of liquid.

Significant figures

- Significant figures = figures needed to represent a measurement without loss of accuracy.
- Example: 0.000304       030.400       23.000        2 x 10^3
  - 3 sig figs        5 sig figs  2 sig figs        1 sig fig
- For logarithms:
  - log (0.000304) = -3.517  (3 sig figs)
  - Characteristic: 3
  - Mantissa: 517

Use of Microsoft Excel

- You are expected to be able to use Microsoft Excel worksheets and graphing capabilities. Please practice by following sections 2-11 and 2-12 in case you have not used MS Excel yet.
- This will be expected of you in your lab reports.

Rules for adding and subtracting

- 23.02521 + 1.2452 x 10^3 becomes
  - 23.02521
  - + 124.52
  - 147.54521 => 147.55
- If it were: 147.54500 then, => 147.54
- (i.e. only round up when it becomes even)
- In general, the answer has the same number of decimal places as the least significant number being added.

Rules of multiplication and division

- (2.305 x 0.0034) / (3.2 + 0.300) =
- Add up the denominator:
- (2.305 x 0.0034) / (3.5) = 7.86 / 3.5 = 2.2
- In general, the final answer does not have more significant figures than any of the factors leading to it.