SYNTHESIS OF POTASSIUM DIHYDROGEN PHOSPHATE (week one)

- the salt potassium dihydrogen phosphate (KH$_2$PO$_4$) will be prepared and its purity determined
  - Develop a understanding of acid-base neutralization reactions
  - Explore acid-base equilibria, including the determination of an equilibrium constant (specifically, $K_{a2}$ for H$_3$PO$_4$)

- Develop skills
  - titration with pH meter to determine equivalence point
  - Use of Mel-Temp (for melting point / purity check)
The synthesis

\[ \text{H}_3\text{PO}_4 \text{(aq)} + \text{KOH} \text{(aq)} \rightarrow \text{KH}_2\text{PO}_4 \text{(aq)} + \text{H}_2\text{O} \text{(l)} \]

- Neutralization of phosphoric acid with KOH\text{(aq)} (abstracting just one H\textsuperscript{+}) produces the soluble compound KH\textsubscript{2}PO\textsubscript{4}
- Removal of the water will leave behind the solid compound that will hopefully
  - be in high yield
  - and with good purity
Understanding the system

<table>
<thead>
<tr>
<th>Ionization Reactions</th>
<th>Equilibrium Constant</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{H}_3\text{PO}_4\text{(aq)} + \text{H}_2\text{O\ (l)} \rightarrow \text{H}_2\text{PO}_4^-\text{(aq)} + \text{H}_3\text{O}^+\text{(aq)}$</td>
<td>$K_{a1} = 6.9 \times 10^{-3}$</td>
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<tr>
<td>production of dihydrogen phosphate</td>
<td></td>
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<tr>
<td>$\text{H}_2\text{PO}_4^-\text{(aq)} + \text{H}_2\text{O\ (l)} \rightarrow \text{HPO}_4^{2-}\text{(aq)} + \text{H}_3\text{O}^+\text{(aq)}$</td>
<td>$K_{a2} = 6.2 \times 10^{-8}$</td>
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<tr>
<td>production of monohydrogen phosphate</td>
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<tr>
<td>$\text{HPO}_4^{2-}\text{(aq)} + \text{H}_2\text{O\ (l)} \rightarrow \text{PO}_4^{3-}\text{(aq)} + \text{H}_3\text{O}^+\text{(aq)}$</td>
<td>$K_{a3} = 4.8 \times 10^{-13}$</td>
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<tr>
<td>production of phosphate</td>
<td></td>
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</tbody>
</table>

All of these systems co-exist and must have a single pH i.e. all have same $[\text{H}_3\text{O}^+]$
pH = -log \([H_3O^+]\)

\[
\begin{align*}
\text{increasing acidity} & \quad \text{neutral} & \quad \text{increasing basicity} \\
pH &= 0 \quad 4 \quad 7 \quad 10 \quad 14 \\
[H^+] &= 10^0 \quad 10^{-4} \quad 10^{-7} \quad 10^{-10} \quad 10^{-14} \\
[OH^-] &= 10^{-14} \quad 10^{-10} \quad 10^{-7} \quad 10^{-4} \quad 10^0
\end{align*}
\]

\[\text{H}_2\text{O} + \text{H}_2\text{O} \Leftrightarrow \text{OH}^- + \text{H}_3\text{O}^+\]

\[K_w = [H_3O^+][OH^-]\]
To shift the composition – simply need to alter the pH.
Titration set-up and meter calibration

- select a pH meter and electrode
  - treat the electrode with care
  - keep it mounted in the electrode holder at all times
  - do not use it as a stirring device
- calibrate the pH meter / probe with buffers
  - As the electrode is transferred from one solution to another, rinse it with distilled water before insertion into the new solution
- the probe may be left in your solution while the titration is performed
The set-up . . .

- Use clamps
- Use stir-bar (magnetic)
- Avoid electrode damage
- Don’t waste time
Calibration . . .

- Clear old calibration
- Use 2 buffers (one at a time!)
- 1\textsuperscript{st} calibration offsets ideal Nernstian curve (does not alter slope)
- 2\textsuperscript{nd} calibration step adjust the slope of the response curve
Calibration curve illustrated. . .
1\textsuperscript{st} Titration (‘‘complete’’ neut. of H\textsubscript{3}PO\textsubscript{4} )

- Obtain 10 mL of H\textsubscript{3}PO\textsubscript{4} (aq) (1.00 M)
- Titrate with KOH (aq) (1.00 M) until \textbf{all} of the acid has been neutralized.
- Record the pH after each 1.00 mL increment of KOH has been added. You may add smaller increments of KOH at each of the equivalence points
  - (hint: at what volumes of added KOH do you expect each successive equivalence point to occur??).
- Select a target pH for the first equivalence point.
25 mL of 1.0 M H₃PO₄ titrated with 1.0 M NaOH
2nd Titration (production of KH$_2$PO$_4$)

- Obtain 25 mL of H$_3$PO$_4$(aq) (1.00 M)
- Titrate with KOH(aq) (1.00 M) until the target pH is reached.
- Record the pH after each 1.00 mL increment is added,
- Add smaller increments as you approach the target pH to avoid passing the equivalence point.
Select target pH for max. \( \text{KH}_2\text{PO}_4 \)

Note: all pH meters / probes will not produce identical data / in fact two trials with the same meter and probe can yield different numbers.
Isolation of the dry product

- Evaporate the solution to near dryness using a hotplate.
- Collect the crystals on a few layers of paper towels and press dry.
- Place the crystals onto a piece of pre-weighed filter paper on a watch glass and allow them to dry completely in your drawer until the following laboratory period when they will be weighed.
- In week two you will characterize the product...
Characterization

- Mass of product (% yield)
- Melting point of product (impurities decrease melting point – colligative property)
- Titration of dihydrogen phosphate ($H_2PO_4^{2-}$) with standardized NaOH solution