SYNTHESIS OF POTASSIUM DIHYDROGEN PHOSPHATE (week two)

- the salt potassium dihydrogen phosphate (KH₂PO₄) has been prepared in week one and allowed to dry

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

Mass of product (% yield)

\[
\text{Theo. Yield} = \frac{\text{volume KOH}}{\text{L sol'n}} \times \frac{\text{mole KOH}}{1 \text{ mole KOH}} \times \frac{1 \text{ mole } \text{KH}_2\text{PO}_4}{1 \text{ mole KOH}} \times \frac{136.086 \text{ g } \text{KH}_2\text{PO}_4}{1 \text{ mole } \text{KH}_2\text{PO}_4}
\]

“stop volume” actual KOH concentration
Select points on our Titration Curve

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**Titration Curve of Phosphoric Acid**

1. **1st equiv. pt.**
   - pH = \( pK_{a1} \)
   - pH = 0.5(\( pK_{a1} + pK_{a2} \))

2. **2nd equiv. pt.**
   - pH = \( pK_{a2} \)
   - pH = \( pK_{a3} \)
   - pH = 0.5(\( pK_{a2} + pK_{a3} \))
# Understanding the system

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

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

- Crude material mass (we will correct this for purity)
  - Purity x (crude mass) = “pure mass”
  - Greater than 100% purity?

- Titration of dihydrogen phosphate ($\text{H}_2\text{PO}_4^{2-}$) with standardized NaOH solution

- Melting point of product (impurities decrease melting point – colligative property)

NOTE: melting point is not in the lab manual procedure – Dr. Mencer added this b/c you did not get to do the alum lab in the fall
Purity Titration

- In week two, weigh ~ 0.002 mole of the crystals (weigh accurately ±0.0001 g),
- dissolve in ~ 25 mL distilled water,
- calibrate your pH meter (same as week one),
- titrate the solution with aqueous NaOH (0.1000 M)* until **all** the acid has been neutralized.
- Measure the pH of the solution frequently and plot the titration curve for submission with your report form.

## Note: if 100% pure KH₂PO₄ exactly 0.002 mole would require 20.00 mL of 0.1000 M NaOH\(_{(aq)} \) for complete neutralization

*Record the actual concentration and the uncertainty: ex. 0.1000 ± 0.0005 M
Melting Point

- Melting point is a crude method of purity determination
- The greater the impurity content the lower the melting point (freezing point depression)
Melting Point

- Sample is loaded in the capillary melting point tube
- Standard is loaded in a separate tube
- With material in the tube, tap on counter (or use “drop tube” method) to force into closed end of capillary
Melting Point

- Pure $\text{KH}_2\text{PO}_4$ melts at a relatively high temperature (look this value up!)
- Do not heat the thermometer above the maximum operating range!
- Read temp. at beginning (onset point), and end (clear point), of the melting process
- Correct the readings by an amount required to …

For more detailed directions visit:
http://orgchem.colorado.edu/Technique/Procedures/Meltingpt/Mel-Temp.html
Melting Point

- Safety Precautions
  - Never insert a room temperature thermometer into a hot Mel-Temp. It may shatter.
  - Do not leave a Mel-Temp on if you are not actually using it, or leave it running unattended.
  - Do not heat the Mel-Temp beyond the upper temperature of the thermometer.
  - Do not touch the heating block – it gets very HOT!

- Reporting the melting range
  - [onset point - clear point]
  - along with the ramping rate (~°C/min),
  - much more reliable than a single number report.