Experiment 2: Determination of the Purity of Potassium Hydrogen Phthalate (KHP)

In this experiment, a sample of KHP of unknown purity will be analyzed by titration with a standardized NaOH solution. The reaction is shown below:

\[
\text{COOH} + \text{OH}^- \rightarrow \text{COO}^- + \text{H}_2\text{O} + \text{K}^+
\]

Note that this is a 1:1 titration. One mole of base will titrate one mole of acid. The endpoint of the titration will be determined by an indicator, phenolphthalein. Phenolphthalein is a weaker acid than KHP. It is colorless under acidic conditions and changes to a pink color under basic conditions. Therefore, the first persistent presence of color indicates that you are beginning to titrate the indicator and have reached the endpoint; if your solution changes to a dark pink color, you have overshot the endpoint.

Procedure:
Remember - Do not put your spatula directly into containers of reagents and do not put your pipette into your bottle of NaOH. Record all data directly into your notebook.

- Clean and label a weighing bottle for your unknown 1 week prior, and place it in the 120° oven! Use an open weighing bottle in a beaker, covered with a watch glass.

1. Remove your unknown from the drying oven and allow it to cool in a desiccator. Do not cap your weighing bottle until your sample has cooled.

2. If necessary, prepare a ~0.1 M NaOH solution and store it in a polyethylene bottle (not glass). You will standardize this solution, so the preparation need not be exact.

3. Put a small amount of dry, primary standard KHP (100%; NOT your unknown) in a clean, dry weighing bottle. Into each of three separate clean 250 mL Erlenmeyer flasks, weigh by difference approximately 0.7-0.9 g of this standard.

4. To each flask, add about 75-100 mL deionized water and swirl gently to dissolve the sample. Add two drops of indicator to each flask.

5. Rinse your burette with your NaOH solution, then fill it. Titrate the standards to the endpoint, carefully recording the initial and final volumes of NaOH for each titration. Make certain that all of the KHP has dissolved before you finish each titration. Your experiment will go faster if you titrate the lightest sample first. Refill the burette before each titration, if necessary.

6. Once your unknown sample has cooled, weigh by difference approximately 1.2-1.5 g of unknown into each of three separate clean 250 mL Erlenmeyer flasks.

7. Repeat steps 4 and 5 for your unknown samples.
8. Perform a blank titration: add 75-100 mL deionized water to a 250 mL Erlenmeyer flask, add two drops of indicator, and titrate. Record the volume required and correct your data if necessary.

**Report:**
on a 3"x5" card or a 1-page spreadsheet, report the following:

- name
- date/day
- title of experiment
- unknown #
- NaOH concentration (M) average/standard deviation
- individual results for the purity of KHP (weight %KHP for each unknown measured)
- average/standard deviation/95% confidence interval (for weight %KHP unknown)
- reaction for the titration

Questions to consider before starting the laboratory (you should enter these in your notebook):
1. Why is a blank necessary?
2. Why must all of the KHP sample be dissolved before reaching the endpoint?
3. Why are the samples dried at 120°C before use? What errors would occur if the samples were not dried?
4. Why do you have to allow your sample to cool before weighing?
5. Suggest a few reasons why the use of Erlenmeyer flasks was specified rather than beakers.
6. Once the titration begins, would rinsing the burette tip with water into the Erlenmeyer flask affect your results? Explain.
7. Why must you prepare the NaOH solution in a plastic container rather than a glass container?