**Acid-Base Titration: Determination of Potassium Hydrogen Phthalate (KHP)**

**Background**

We review basic titration techniques via a simple base standardization and then titration of an unknown solid containing Potassium Hydrogen Phthalate, commonly abbreviated as KHP. The molecular formula for Potassium Hydrogen Phthalate is KHC8H4O4 and its molecular mass is 204.2212 grams/mole. The base used in this titration (sodium hydroxide, NaOH) is prepared *approximately* and then standardized via titration with a primary standard (KHP in this experiment). A primary standard is a highly pure substance that also must be stable (@ room or elevated temperatures) and not hygroscopic in nature. The standardized base is used to then determine the percent composition of an unknown containing KHP. Useful equations for this experiment are located in the Proper Use of Lab Equipment handout.

**Prepare 0.1 M Sodium Hydroxide Solution**

Calculate the amount of solid NaOH (NaOH = 40 g/mole) needed to make 2 L of ~0.1 M NaOH. Add 500 mL of Nanopure H2O to a 1 L volumetric flask and add the NaOH to the water. Completely dissolve the NaOH, and then dilute the solution to the 1 L mark. This solution should be transferred to the 2 L glass storage bottle with the rubber stopper. Using a volumetric flask, add an additional 1 L of H2O to the storage bottle, stopper, and shake vigorously. The NaOH does not need to be weighed precisely. This NaOH solution is used in this experiment and potentially two others. Any remaining NaOH solution can be saved for the later labs.

**Standardize Sodium Hydroxide against Potassium Hydrogen Phthalate (KHP)**

Potassium hydrogen phthalate is a white, crystalline solid, which is suitable for use directly as ***a PRIMARY STANDARD***. It is best to dry KHP for a few hours in an oven at 100oC to remove traces of moisture before using it (TA will have placed the pure KHP in the oven for you). It is then best stored in a desiccator.

**KHC8H4O4 + NaOH 🡺 KNaC8H4O4 + H2O**

KHC8H4O4 is the first potassium salt of phthalic acid (benzene-1,2-dicarboxylic acid).

Obtain a quantity of dried, pure KHP in a glass weighing bottle/top and keep it stored in your desiccator.

Weigh a 0.7-0.8 g sample (to the nearest 0.1mg i.e., 0.0001 g) of KHP. Use an analytical balance to complete weighing, recording all significant digits reported. Make sure the units of the analytical balance are grams! It is best if the samples of KHP are weighed by difference, but not necessary. The use of weigh boats or weighing paper is allowed as long as KHP aliquot is quantitatively transferred into a 250 mL Erlenmeyer flask. Once the KHP aliquot is transferred to the 250 mL Erlenmeyer flask, dissolve it in 50-75 mL of Nanopure H2O measured using a 100.0 mL graduate cylinder.

Add 3 drops of the phenolphthalein indicator to the KHP solution in the 250 mL Erlenmeyer flask. Titrate with base until a veryfaint pink color remains for at least 30 seconds (the endpoint). An example of endpoint and over-titrated point will be presented during pre-lab lecture. Record the volume of base used in the titration using the correct significant figures for a buret reading, two digits past the decimal. Calculate the concentration [molarity] of the NaOH solution. Repeat the titration process at least two more times and calculate the average, standard deviation, and ppt (parts per thousand) values for the molarity of NaOH. **Your results should be within 5 ppt**. The ppt equation is found at the end of Proper Use of Lab Equipment handout. If results are not consistent/within 5ppt, it is suggested to complete a 4th or 5th titration.

**Determine Percent KHP in an Unknown Sample**

Obtain a vial of an Unknown KHP sample from the Teaching Assistant (it is dried in an oven ahead of time). Put your signature on the unknown sign-up sheet, next to the unknown number chosen [found on the unknown vial]. To cool & keep the unknown sample dry, keep the unknown vial in a silver desicooler.

To determine the percent composition of the unknown, calculate the appropriate amount of unknown [using the appropriate amount equation] needed to titrate an endpoint (faint pink) using *about* 30.00 mL of the standardized NaOH solution. Weigh out an aliquot of unknown sample to the nearest 0.1 mg using an analytical balance and quantitatively transfer it into a 250 mL Erlenmeyer flask. Dissolve the unknown in 50 - 75 mL of Nanopure H2O. Then, add 3 drops of phenolphthalein indicator to the 250 mL Erlenmeyer flask and titrate with NaOH to the faint pink endpoint. Record the volume of base used to reach the endpoint, recording the correct significant digits for a buret reading. NOTE: This titration will most likely NOT use 30.00 mL of NaOH to titrate to the endpoint.

As the previous titration most likely did not require at least 30.00 mL or thereabouts of NaOH, calculate the new, adjusted mass of unknown sample needed to titrate/reach the endpoint for the unknown with about 30.00 mL of standardized NaOH solution. Repeat the titration at least two more times using the new adjusted mass of unknown recording all necessary data. Calculate the percent composition of KHP for each trial. Then calculate the average % KHP, standard deviation, and ppt of the unknown KHP. **Your results should be within 5 ppt**. If this is not the case, it is suggested to complete a 4th or 5th titration. If the solid unknown sample in the vial is running low, alert your Teaching Assistant as extra unknowns must be dried *at least 24 hours* before being provided to you. Unknown KHP percentages are in the range of 27.00 - 60.00 %.