Chemistry 119: Experiment 3

Preparation of a Standard Sodium Hydroxide Solution
and Titration of Hydrochloric Acid

In this experiment, we prepare solutions of NaOH and HCl which will be used in later experiments. We will require a knowledge of the exact concentration of the two solutions, but it is not convenient either to weigh out solid NaOH or to measure out concentrated HCl. We do not know the composition of these materials well enough to obtain accurate numbers for our solutions by weighing them out.

Instead, we take another approach. We will use the **primary standard** acid, potassium hydrogen phthalate, to make an acid solution containing a known amount of acid. The composition and purity of this acid is very well known, and we can accurately determine the amount of potassium hydrogen phthalate by weighing a sample. We can titrate this solid acid, after dissolving it in water, with our NaOH solution to accurately determine the NaOH concentration, as discussed below. This **standardizes** the NaOH solution against our primary standard. Then, with our standardized NaOH solution, we can titrate the HCl solution and accurately determine the HCl concentration. Thus, the concentrations determined for the NaOH and HCl solutions ultimately depend on the weight of our primary standard taken and the volumes of the solutions needed to react. Because the NaOH and HCl will be used to analyze other materials in future weeks, it is very important to determine their concentrations as carefully as possible. This suggests that careful weighing of the potassium hydrogen phthalate is very important.

The presence of carbon dioxide is one reason why we cannot weigh out pure NaOH and use it as a primary standard. Water which is in contact with the atmosphere can readily absorb carbon dioxide. Dissolved carbon dioxide acts as a weak acid in aqueous solutions, as shown in the following equation.

$$CO_2\ (g)\ +\ H_2O\ =\ HCO_3^-\ +\ H^+ \tag{4.1}$$

If basic solutions are prepared using water which contains dissolved carbon dioxide, a portion of the base reacts with the carbon dioxide to yield hydrogen carbonate and carbonate. For this reason, we will need to use water from which all carbon dioxide has been removed to prepare any sodium hydroxide solutions which will be standardized.

Another reason why standard sodium hydroxide solutions cannot be prepared by weighing is that the solid reagent often contains significant amounts of impurities, and is highly hygroscopic. Usually, sodium hydroxide solutions are prepared by diluting a 50% aqueous solution of sodium hydroxide to approximately the desired concentration, followed by standardization of the solution by titration of an acidic

primary standard. In 50% solutions of sodium hydroxide, dissolved carbon dioxide precipitates as sodium carbonate.

In this experiment potassium hydrogen phthalate ($C_8H_5O_4K$, called KHP for short) is used as the primary standard. The KHP standard is a stable, pure, well-characterized material which slowly dissolves in water to produce K^+ and hydrogen phthalate ($C_8H_5O_4^-$) ions. KHP contains one titratable acidic hydrogen ($pK_a = 4.01$), which reacts according to the equation

$$\qquad\qquad + \ OH^- \ \rightleftharpoons \qquad\qquad + \ H_2O \qquad\qquad (4.2)$$
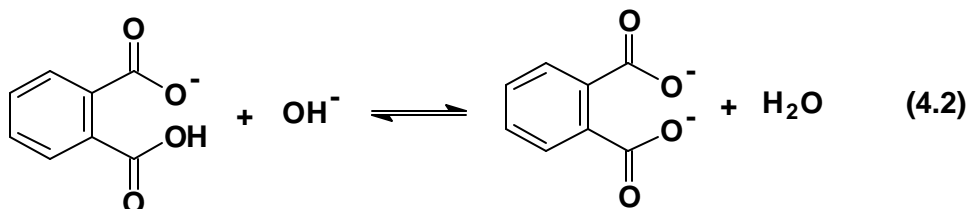
Prelaboratory Assignment

A 0.5123-g sample of KHP (F.W. 204.23) was dissolved in about 25 mL of distilled water, and titrated to the phenolphthalein end point with 28.75 mL of a sodium hydroxide solution. Calculate the molar (M) concentration of the hydroxide solution.

Apparatus
3 250-mL Erlenmeyer flasks
50-mL burette
10-mL graduated cylinder
100-mL graduated cylinder
25-mL pipette
2 1-liter polyethylene bottles or glass
   bottles with screw caps
weighing bottle for drying KHP

Chemicals
hydrochloric acid (12 M)
phenolphthalein solution (0.1% in ethanol)
potassium hydrogen phthalate solid (reagent-grade)
50% sodium hydroxide solution (or use 50 g sodium hydroxide in 50 mL of water)

Procedure

1.   Dry the potassium hydrogen phthalate in a 105 °C oven for at least one hour. Use your weighing bottle, without lid, to hold the solid. Label a 150-mL beaker, put the weighing bottle in the beaker and cover the beaker with a watch glass.

2.   While the KHP is drying, clean your burettes and 1-liter bottles. First use tap water, then give a final rinse with deionized water.

3.   Add <u>about</u> 6 mL (use a 10-mL graduated cylinder) of the 50% sodium hydroxide solution to a 1-liter bottle. Fill the bottle to its shoulder with deionized water. Place the stopper or the lid on the bottle, and thoroughly mix the solution by shaking the bottle.

> **Warning**: Concentrated NaOH is very *corrosive*! Handle this solution very carefully, and avoid contact with your skin or clothes. If you do spill some, wash it off at once. Wash your hands carefully when you complete this laboratory.

4.   After having dried it in the oven for at least one hour, place the KHP in a desiccator to cool to room temperature. This usually requires about 15 minutes. Weighing a warm solid will introduce substantial errors, so be sure that the solid is cool.

5.   Weigh three 0.4- to 0.7-g samples to the nearest 0.1 mg. Place each sample in a labeled, 250-mL Erlenmeyer flask. Add about 30 mL of the deionized water to each flask, and dissolve the KHP.

6.   Add two or three drops of phenolphthalein solution to each flask, fill the 50-mL burette with sodium hydroxide solution, and titrate the KHP solution in each flask to the end point. At the end point the solution changes from colorless to a faint pink.

7.   Discard the titrated solutions in each of the three Erlenmeyer flasks, and rinse the flasks first with tap water, and then with distilled or deionized water.

[THIS COMPLETES THE STANDARDIZATION OF YOUR NaOH SOLUTION. BE CERTAIN YOUR STORAGE BOTTLE IS LABELED WITH YOUR NAME AND SECTION. YOU WILL USE THIS SOLUTION IN FUTURE EXPERIMENTS].

8.   Obtain about 8 mL of concentrated hydrochloric acid and dilute to about 1 liter. Mix well, then place in a 1-liter glass bottle or in a 1-liter polyethylene bottle for storage. Label the bottle with your name and section. The hydrochloric acid now has a concentration near 0.1 M.

> **Warning**: Concentrated HCl is very *corrosive* and has *toxic* vapors! Handle this acid very carefully, and avoid contact with your skin or clothes. If you do spill some, wash it off at once. Wash your hands carefully when you complete this laboratory.

9.   Pipette 25 mL of the hydrochloric acid sample into each of the three 250-mL flasks, and dilute with <u>about</u> 25 mL (use a graduated cylinder) of the deionized water.

10.   Add two or three drops of phenolphthalein to each flask, and titrate each solution to the end point with the sodium hydroxide solution.

11.   STORE THE HYDROCHLORIC ACID SOLUTION IN THE PLACE INDICATED BY THE TEACHING ASSISTANT, AGAIN WITH YOUR NAME ON THE BOTTLE. You are depending on the stability of both the NaOH and HCl solutions, so be sure to tighten the caps well!

<u>Calculations and Report</u>.

1.   Use the mass of KHP added to each flask, and the volume of sodium hydroxide solution used in each of the three standardization titrations to calculate the concentration of sodium hydroxide used in each titration.

2.   Calculate the average sodium hydroxide concentration and the standard deviation of the results.

3.   Use the average sodium hydroxide concentration, the end-point volume for each titration, and the volume of the hydrochloric acid sample used in each titration to calculate the concentration of the hydrochloric acid sample.

4.   Calculate the average hydrochloric acid concentration and the standard deviation of the results.

5.   Check your calculations for errors. Is your answer <u>reasonable</u>?

6.   Phenolphthalein (transition pH range 8.0-9.6) is very appropriate for the titration of KHP with NaOH. Why? Other indicators with transition ranges reaching as low as 6 could be used for the HCl /NaOH titration. Why?

7.   In your report, include all data, all the calculations described above and answer the questions. Report the average concentrations found for the sodium hydroxide and hydrochloric acid solutions as well as the standard deviations and the relative standard deviations for both standardizations.

This experiment has been adapted from a laboratory manual authored by Professor S. D. Brown