

## Preparation of a NaOH Standard Solution using Direct Titration

This experiment demonstrates the most common method for obtaining standard solutions for titrimetric analysis. It involves preparation of a solution that has the approximate concentration desired (usually within 10%), determination of the concentration by *direct titration* against a *primary standard*, and a test of the accuracy of your determined concentration by comparison with a known standard. It is important to standardize your solution carefully, as it will be used in later experiments. You should be able to determine your NaOH concentration to  $\pm 0.5\%$  of its actual value. You will be graded on your accuracy.

### Required Reading

D.C. Harris, *Quantitative Chemical Analysis* (9 th ed., W. H. Freeman, NY, 2016) pp. 145-148, 215-217,

Electronic Balances (including Weighing by Difference), Analytical Lab Manual  
Burets, Analytical Lab Manual

In addition to being able to explain the purpose of your experiment, the general procedure steps, the use of all chemicals in this experiment and any specific hazards, your prelab quiz may include explanations of any of the following terms: buret, titration, primary standard, equivalence point, endpoint, indicator, standardization. You should know all relevant chemical reactions for this experiment. Describe the chemical and/or physical processes that will occur when you reach the endpoint of your titration. What characteristics of KHP make it a good primary standard?

### Chemicals and their Location

*Balance Room*

Potassium hydrogen phthalate

*Fume Hood*

Phenolphthalein, indicator soln

Sodium hydroxide, ~50 % w/v aqueous soln

Hydrochloric acid, vol. std.

### Equipment and its Location

*Your Drawer*

Buret

Weighing bottle

### Safety Issues and Chemical Hazard Information

	<i>Physical Hazards</i>	<i>Health Hazards</i>
<i>Hydrochloric acid</i>	water-reactive, corrosive	toxic
Phenolphthalein	none	irritant
<i>Potassium hydrogen phthalate</i>	none	irritant
<i>Sodium hydroxide</i>	water-reactive, corrosive	toxic, irritant

## Procedure

There are various components to this procedure. If there is a delay at one step (e.g., all the balances are in use), perform another section of this procedure while you are waiting.

### *Preparation of a Primary Standard (KHP)*

1. KHP is a primary standard. It is pre-dried and kept in desiccators in the balance room. To minimize contamination, use the spatula provided in the desiccator — do not use your own spatula. Using a clean dry weighing bottle, weigh accurately by difference, triplicate  $\sim 0.8$  g samples of KHP, into labeled 250 mL Erlenmeyer flasks. To each flask, add 75 mL of DI water (measure by graduated cylinder) and three drops of phenolphthalein indicator. Swirl gently. Cover the flasks and leave to dissolve, swirling periodically.

### *Preparation of Approximate Solution (0.1M NaOH)*

2. Using the 50 % NaOH Solution provided, calculate (for 1 L) and prepare your  $\sim 0.1$  M NaOH solution, by adding the calculated amount of 50 % solution to one of your 1 L plastic bottles (half filled with freshly boiled DI water). Once the concentrated NaOH has been added, fill the bottle up to the neck with the boiled NaOH. Clean your buret, and rinse with a small aliquot of your NaOH solution, before filling it with the NaOH solution. Make a note of the volume of liquid in your buret within 0.02 mL precision.

### *Standardization of NaOH with KHP*

3. Begin to titrate your first KHP solution by adding NaOH rapidly until a pink color is noticed. A piece of white paper under the titration flask will aid in observing the color change. At this point, slow the addition of NaOH so that only a localized pink color is observed in the stirred solution. Continue slowing the rate of addition to keep the amount of pink color localized. Stop every so often to be sure that all color disappears upon mixing. As the endpoint is approached, a single drop of NaOH may turn the solution pink for a moment. Proceed very slowly with drop by drop additions until the entire solution remains light pink for at least 60 seconds and after heating to the boil. If the color fades add one drop more of the NaOH soln. At this point the endpoint has been reached. Record the final volume of the buret (to 2 decimal places). Similarly, titrate your other two flasks of KHP. The correct endpoint is a clear solution with a faint pink tinge. If your solution is hot pink, you have passed the endpoint. Try to do better on your other replicate measurements.

**Be careful** not to let the fluid level in the buret drop below the 50 mL line. If more than 50 mL will be required for a titration, stop the titration before reaching the 50 mL line, record the exact volume, refill the buret, record the new initial volume, and continue with the titration. Upon completion, the two volumes can be added to obtain the total titrant volume.

### *Comparison of NaOH solution to HCl Volumetric standard*

4. To each of three clean labeled 250 mL Erlenmeyer flasks, add 50 mL of DI water and 3 drops of phenolphthalein. Fill your other buret with the standard HCl soln. Record the initial reading of the HCl buret to the nearest 0.01 mL. Add approximately 35, 40 and 45 mL of the acid to flasks #1, #2 and #3, respectively, recording the initial and final buret readings each time, so that you can calculate exactly how much acid has been added to each flask. Titrate the HCl with your NaOH solution in the same way you titrated the KHP.

### *Shutdown procedures*

**IMPORTANT NOTE:** Your NaOH solution must be saved upon completion of the experiment; you will use it again in future experiments.

5. Clear up your work area. Return items to the Fume hood, etc.

### **Data Analysis**

Read the Report Format: *Data Analysis* and the *Useful Analysis Techniques: Data reporting* in your lab reports sections of this manual before working on your first Data Analysis and make sure you follow them.

1. Using the weight of KHP in each flask, and the molecular weight of KHP, calculate the number of moles of KHP in each flask.
2. Calculate the molarity of your NaOH solution using the total volume of NaOH used to titrate each KHP flask to its endpoint. (Don't forget to convert your volume from mL to liters.) Report your standardized NaOH concentration as the average of your three replicates with appropriate statistical analysis (see *Report Format: Results section*).
3. Using your experimentally determined standardized NaOH concentration (use the average value), determine the molarity of your volumetric standard HCl. Report your determination of the HCl concentration as the average of your three replicates with appropriate statistical analysis (see *Report Format: Results section*).

### Discussion Questions

1. Why can't we just dissolve a known mass of NaOH in a known volume of water and calculate the NaOH concentration? In other words, explain why you standardized your NaOH solution.
2. Ideally, when performing aqueous acid-base titrations, all DI water should be boiled prior to use to remove dissolved carbon dioxide. All solutions should then be kept tightly closed when not in use; NaOH solutions are especially capable of adsorbing large amounts of CO<sub>2</sub> from the atmosphere. Why would we ideally want to eliminate CO<sub>2</sub> from solutions when performing acid-base titrations?
3. Describe how you would weigh your KHP samples, if you didn't know about weighing by differences. Mentioning any differences between your method and the method we made you use, explain why we use the technique of weighing by difference.