

Preparation of an 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* (7th ed., W. H. Freeman, NY, 2007) pp. 121-124, 221–218,

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
<i>Phenolphthalein</i>	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 predried 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 ~0.8 g samples of KHP, into labeled 250 mL Erlenmeyer flasks. To each flask, add 75 mL of distilled 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 ~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 colour is noticed. A piece of white paper under the titration flask will aid in observing the colour change. At this point, slow the addition of NaOH so that only a localized pink colour is observed in the stirred solution. Continue slowing the rate of addition to keep the amount of pink colour localized. Stop every so often to be sure that all colour 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 distilled water and 3 drops of phenolphthalein. Fill your other buret with the standard HCl soln. Record the initial reading of the HCl burette 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 burette 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.
6. Make sure you have recorded information on the sample you studied, in this case, the number/letter identifying the HCl volumetric sample, and the number/letter identifying your NaOH starting solution.

Waste Disposal

Waste Chemicals	
All neutralized, and dilute acid/base solutions	
KHP solid	Solid KHP
NaOH	Base

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 distilled 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 CO₂ from the atmosphere. Why would we ideally want to eliminate CO₂ from solutions when performing acid-base titrations? You did not use boiled distilled water to make your KHP solution. What effect might this have on your standardization – would it raise or lower the concentration you determined for your NaOH solution? We do not take particular care storing your NaOH solution after it has been standardized. If you used it to determine an unknown acid at the end of the semester, what effect might the absorbed CO₂ have on your determination?
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.